# 410 Rec'd PCT/PTO 1 7 SEP 2001

FORM PTO-1390 U.S. DEPARTMENT OF COM	IMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER	
TRANSMITTAL LETTER TO THE UNITED STATES		02365	
DESIGNATED/ELECTED OFFICE (DO/EO/US)		U.S. APPLICATION NO. (If known, see 37 CFR 1 5	
CONCERNING A FILING UNDER 35 U.S.C. 371		09/936756	
INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED	
PCT/EP00/02258	15 March 2000 (15.03.00)	15 March 1999 (15.03.99)	
TITLE OF INVENTION METHOD FOR	PRODUCING PHYSICALLY FOAME	ED INJECTION MOULDED PARTS	
APPLICANT(S) FOR DO/EO/US ULRIC			
Applicant herewith submits to the United St	tates Designated/Elected Office (DO/EO/US)	the following items and other information:	
1. X This is a FIRST submission of items	s concerning a filing under 35 U.S.C. 371.		
2. This is a SECOND or SUBSEQUE	NT submission of items concerning a filing	under 35 U.S.C. 371.	
This is an express request to begin items (5) (6) (9) and (21) indicated	national examination procedures (35 U.S.C. below.	371(f)). The submission must include	
4. The US has been elected by the exp	piration of 19 months from the priority date (	(Article 31).	
5. X A copy of the International Applica	tion as filed (35 U.S.C. 371(c)(2))	ional Rureau)	
a. is attached hereto (require	ed only if not communicated by the Internat	ionai Buicau).	
b. X has been communicated by	by the International Bureau. Dication was filed in the United States Receive	ving Office (RO/US).	
	the International Application as filed (35 U.S.		
An English language translation of $X$ a. $X$ is attached hereto.	me memanona approacon as mea (55 c.)		
a. X is attached hereto.  b. has been previously subm	nitted under 35 U.S.C. 154(d)(4).		
Amendments to the claims of the Ir	nternational Aplication under PCT Article 19	(35 U.S.C. 371(c)(3))	
a. are attached hereto (requi	ired only if not communicated by the Interna		
b. have been communicated	by the International Bureau.		
c. have not been made; how	— — — — — — — — — — — — — — — — — — —		
d. have not been made and will not be made.			
An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).			
9. An oath or declaration of the inve			
An English lanugage translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).			
Items 11 to 20 below concern docum	ent(s) or information included:		
	ment under 37 CFR 1.97 and 1.98.		
	ording. A separate cover sheet in compliance	be with 37 CFR 3.28 and 3.31 is included.	
13. A FIRST preliminary amendment 14. A SECOND or SUBSEQUENT			
<u> </u>			
15. A substitute specification.  16. A change of power of attorney a	and/or address letter.		
	e sequence listing in accordance with PCT R	ule 13ter.2 and 35 U.S.C. 1.821 - 1.825.	
	international application under 35 U.S.C. 15		
	anguage translation of the international applic	Canon under 35 O.S.O. 157(4)(7).	
20. \( \overlight\) Other items or information:			
English translation of the application as amended. International Preliminary Examination Report including amendments (in German)			
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1.137 (a) or (b))	must be filed an	d grante	ed to restore the applica	tion to pending state		11
SEND ALL CORRESPONDENCE TO:						
Michele J. Young			7 1			
Salter & Michaelson			I U			
321 South Main Street Providence, RI 02903-7128  Michele J. Young NAME						
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# ATTORNEY DOCKET NO. 02365 IN THE U.S. PATENT AND TRADEMARK OFFICE

Applicant: Ulrich STIELER

Serial No. : Unknown

Filed : Herewith

Title : METHOD FOR PRODUCING PHYSICALLY FOAMED INJECTION

MOULDED PARTS

Assistant Commissioner for Patents Washington DC 20231

# PRELIMINARY AMENDMENT

#### Sir/Madam:

Prior to examination of the above-identified application, entry of this preliminary amendment is respectfully requested. Please amend the above-identified application as follows:

# In the Specification:

Please amend the specification as follows:

Please replace the specification with the substitute specification, excluding claims, submitted herewith under 37 C.F.R. 1.125(b).

# In the Claims:

Please rewrite the claims as follows:

1. (Amended Once) A process for the production of physically foamed injection molded articles, wherein in a first stage a propellant-free first melt portion is fed into a cavity (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion

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(propellant injection phase), and possibly in a third stage a propellant-free further melt portion is charged into the cavity, the production of the injection molded articles occurring in the cavity,

wherein metering of the physical propellant in the second stage occurs in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure which is exerted on the propellant in the phases between or before or after metered addition, and the expansion of the propellant occurs in the cavity.

- 2. (Amended Once) The process of Claim 1, wherein the propellant is a compressible fluid.
- 3. (Amended Once) The process of Claim 1 further comprising the step of maintaining the propellant under pressure in the intermediate cycle times before and after the propellant injection phase.
- 4. (Amended Once) The process of Claim 3, further comprising maintaining the propellant at a pressure of at least p (crit) at a given temperature during the intermediate cycle times.
- 5. (Amended Once) The process of Claim 1, further comprising the step of controlling the pressure exerted on the propellant via a pressure control valve.
- 6. (Amended Once) The process of Claim 5, wherein the pressure control valve is a multi-way valve.
- 7. (Amended Once) The process of Claim 6, wherein the multi-way valve is a 3/3-way proportional valve or a 2/3-way proportional valve.

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8. (Amended Once) The process of claim 1 further comprising the step of controlling the

pressure of the critical propellants via at least one pressure relief valve connected downstream of the

pressure control valve.

9. (Amended Once) The process of Claim 8, wherein at least one of the pressure relief valves

has a holding pressure equal to or higher than the pressure at which a critical propellant is held in

the intermediate cycle times.

10. (Amended Once) The process according to Claim 1 further comprising the step of regulating

the pressure preset by the pressure control valve via one or more pressure relief valves to the

injection pressure at which the propellant is added to the melt via an injection point.

11. (Amended Once) The process of claim 1, wherein the injection point is configured as a

throttle means.

12. (Amended Once) The process of Claim 11, wherein the injection point is in the form of a

defined gap in an injector or of an injector with a sinter metal.

13. (Amended Once) The process of Claim 11, wherein the injection point is configured as a

controlled closure mechanism.

14. (Amended Once) The process of Claim 1 further comprising the step of using water as the

propellant.

15. (Amended Once) The process of Claim 1 further comprising the step of using a gas or gas

mixture as the propellant.

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16. (Amended Once) The process of Claim 15, further comprising the step of using carbon

dioxide as the propellant.

17. (Amended Once) The process of Claim 16, wherein the carbon dioxide is held in the

intermediate cycle times at a pressure of at least 60 bar.

18. (Amended Once) The process of Claim 1 further comprising the step of elevating the

pressure of the propellant during the propellant injection phase to a pressure of over 60 bar using

the pressure control valve.

19. (Amended Once) The process of Claim 1 further comprising the step of generating a

counterpressure in the cavity.

20. (Amended Once) The process of Claim1, wherein the physically foamed injection molded

article is selected from the group consisting of a handle, a knob, a gearshift knob, a steering wheel

casing, a ball, a sphere, a fender, a float and a closing means for bottle-like containers.

21. (Amended Once) A device for the metered addition of physical propellants to a foamable

melt, comprising:

a storage means, in which the propellant is stored under pressure,

a pressure control valve for regulating the propellant pressure, and

an injection point, which is configured as a throttle means, at which the propellant under

pressure is fed to the melt,

wherein a controlled closure mechanism is provided at the injection point.

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22. (Amended Once) The device of Claim 21, further comprising at least one pressure relief valve.

Please add the following claims:

--23. (New) The process of claim 1, further comprising the step of:

maintaining the propellant in a compressed state in the intermediate cycle times
before and after the propellant injection phase.

24. (New) A device for the metered addition of physical propellants to a foamable melt, comprising:

a storage means, in which the propellant is stored under pressure,

a pressure control valve for regulating the propellant pressure, and

an injection point, which is configured as a throttle means, at which the propellant under pressure is fed to the melt,

wherein at least one pressure relief valve is provided at the injection point.--

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#### **REMARKS**

By way of this Preliminary Amendment, the English translation of the Specification has been amended to conform to U.S. Practice. A Substitute Specification excluding claims under 37 C.F.R. 1.125(b) is submitted herewith accompanied by a marked-up copy of the specification showing the matter being added to and the matter being deleted from the specification of record. The Substitute Specification does not include new matter.

In addition, by the present amendment, claims 1-22 have been amended to conform to U.S. Practice. These amendments are not considered to narrow the scope of the claims. Claims 23 and 24 have been added, and are directed to alternative elements of original claims 3 and 22.

The Applicant respectfully submit that no new matter has been added by this Preliminary Amendment, and respectfully requests entry of this preliminary amendment.

#### **CONCLUSION**

In view of the foregoing amendments and remarks, the Applicant respectfully submits that the pending claims in the above-identified application are in condition for allowance, and a notice to that effect is earnestly solicited.

If the present application is found by the Examiner not to be in condition for allowance, then the Applicant hereby requests a telephone or personal interview to facilitate the resolution of any remaining matters. Applicant's attorney may be contact by telephone at the number indicated below to schedule such an interview.

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The Patent and Trademark Office is authorized to charge any additional fees incurred as a result of the filing hereof or credit any overpayment to our Deposit Account No. 19-0120.

Respectfully submitted, STIELER, Ulrich, Applicant

By:

Michele J. Young, Reg/1

Applicant's Attorney

SALTER & MICHAELSO

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Fax: (401) 861-1953 Customer No.: 000987

Dated: September 17, 2001

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# Version with marking to show changes made:

1. (Amended Once) A [P]process for the production of physically foamed injection [moulded]  $\underline{molded}$  articles, wherein in a first stage a propellant-free first melt portion [(6)] is fed into a cavity [(1)] (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), and possibly in a third stage a propellant-free further melt portion is charged into the cavity [(1)], the production of the injection [moulded]  $\underline{molded}$  articles occurring in the cavity,

[characterised in that] wherein metering of the physical propellant in the second stage occurs in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure which is exerted on the propellant in the phases between or before or after metered addition, and the expansion of the propellant occurs in the cavity [(1)].

- 2. (Amended Once) <u>The [P]process [according to] of Claim 1, [characterised in that] wherein the propellant is a compressible fluid.</u>
- 3. (Amended Once) <u>The [P]process [according to] of Claim 1 [or 2, characterised in that] further comprising the step of maintaining the propellant [is kept] under pressure in the intermediate cycle times before and after the propellant injection phase[, or is present in a compressed state].</u>
- 4. (Amended Once) The [P]process [according to] of Claim 3, [characterised in that in] further comprising maintaining the propellant at a pressure of at least p (crit) at a given temperature during the intermediate cycle times [the propellant is held a pressure of at least p (crit) of the propellant at the given temperature].

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5. (Amended Once) <u>The [P]process [according to one of the preceding claims] of Claim 1,</u> [characterised in that] <u>further comprising the step of controlling</u> the pressure exerted on the

propellant [is controlled] via a pressure control valve [(10)].

6. (Amended Once) <u>The [P]process [according to] of Claim 5, [characterised in that] wherein</u>

the pressure control valve [(10)] is a multi-way valve.

7. (Amended Once) The [P]process [according to] of Claim 6, [characterised in that] wherein

the multi-way valve is a 3/3-way proportional valve or a 2/3-way proportional valve [is used as

multi-way valve].

8. (Amended Once) The [P]process [according to one of the preceding claims, characterised in

that of claim 1 further comprising the step of controlling the pressure [control in the case] of the

critical propellants [additionally occurs] via at least one pressure relief valve [(4) which is] connected

downstream of the pressure control valve [(10)].

9. (Amended Once) The [P]process [according to] of Claim 8, [characterised in that] wherein

[the holding pressure of] at least one of the pressure relief valves [(4) is] has a holding pressure equal

to or higher than the pressure at which a critical propellant is held in the intermediate cycle times.

10. (Amended Once) The [P]process according to [one of the preceding claims, characterised in

that Claim 1 further comprising the step of regulating the pressure preset by the pressure control

valve [(10) is regulated] via one or more pressure relief valves [(4)] to the injection pressure at which

the propellant is added to the melt via an injection point [(5)].

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11. (Amended Once) <u>The [P]process [according to one of the preceding claims, characterised in </u>

that] of claim 1, wherein the injection point [(5)] is configured as a throttle means.

12. (Amended Once) The [P]process [according to] of Claim 11, [characterised in that] wherein

the injection point [(5)] is in the form of a defined gap in an injector or of an injector with a sinter

metal.

13. (Amended Once) The [P]process [according to one of Claims 11 or 12, characterised in that]

of Claim 11, wherein the injection point [(5)] is configured as a controlled closure mechanism.

14. (Amended Once) The [P]process [according to] of Claim 1[ or one of the preceding Claims

3 to 13, characterised in that] <u>further comprising the step of using</u> water [is used] as <u>the propellant</u>.

15. (Amended Once) The [P]process [according to one of the preceding Claims 1 to 13,

characterised in that of Claim 1 further comprising the step of using a gas or gas mixture [is used]

as the propellant.

16. (Amended Once) The [P]process [according to] of Claim[s] 15, [characterised in that] further

comprising the step of using carbon dioxide [is used] as the propellant.

17. (Amended Once) The [P]process [according to] of Claim[s] 16, [characterised in that]

wherein the carbon dioxide is held in the intermediate cycle times at a pressure of at least 60 bar [(=

p (crit)  $CO_2$  at room temperature)].

18. (Amended Once) The [P]process [according to one of the preceding claims, characterised in

that for of Claim 1 [the propellant injection phase] further comprising the step of elevating the

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pressure of the propellant [is brought] <u>during the propellant injection phase</u> to a pressure of over 60 bar [via] <u>using</u> the pressure control valve [(10)].

- 19. (Amended Once) The [P]process [according to one of the preceding claims, characterised in that] of Claim 1 further comprising the step of generating a counterpressure [is generated] in the cavity [(1)].
- 20. (Amended Once) <u>The [P]process [according to one of the preceding claims, characterised in that] of Claim1, wherein the physically foamed injection [moulded] molded article is selected from the group consisting of a handle, a knob, a gearshift knob, a steering wheel casing, a ball, a sphere, a fender, a float and a closing means for bottle-like containers.</u>
- 21. (Amended Once) <u>A</u> [D]<u>d</u>evice for the metered addition of physical propellants to a foamable melt, [wherein the device comprises] <u>comprising:</u>
  - a storage means [(11)], in which the propellant is stored under pressure,
  - a pressure control valve [(10)] for regulating the propellant pressure, and
- an injection point [(5)], which is configured as a throttle means, at which the propellant under pressure is fed to the melt, [characterised in that]

wherein a controlled closure mechanism is provided at the injection point [(5)].

22. (Amended Once) The [D]device [for the metered addition of physical propellants according to] of Claim 21, [characterised in that instead of the controlled closure mechanism or in addition to the controlled closure mechanism,] further comprising at least one pressure relief valve [(4) is provided].

U9/936/56

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Marked up Copy of Substitute
Substitute
Specification

Process for the Production of Physically Foamed Injection Moulded Articles

The present invention

# PROCESS FOR THE PRODUCTION OF PHYSICALLY FOAMED.

# INJECTION MOLDED ARTICLES

#### <u>BACKGROUND</u>

# Technical Field

The present disclosure relates to a process for the production of physically foamed injection moulded articles and, in particular, to a process for the production of physically foamed injection moulded articles with an internal foam structure and a compact closed-pore external skin of the same material as the base body.

# Related Art

The production of foamed plastics, for example, is achieved with the aid of so-called propellants, which expand a plastic, generally thermally softened plastic mass in the desired manner. In this case, the propellants are either generated in situ via chemical reaction of the components (chemical propellants), or compressed fluids, e.g.  $N_2$ ,  $CO_2$ , are added under pressure to the starting material, in which case a foaming process of the plastic mass caused by the propellant is initiated upon the subsequent drop in pressure of the component mixture to normal pressure.

However, chemical propellants have a series of disadvantages. For instance, for use in foam injection moulding higher temperatures than are actually necessary for softening starting materials may have to be selected in order to reach the ignition point of the propellants, since the temperature at which the reaction of the components generating propellant starts is generally very high. Because of the high temperatures a higher expenditure of energy is necessary during melting of the raw materials. In addition, the cycle or cooling times are increased and a higher cooling power of the

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cooling plants is necessary. In some circumstances, damage to the raw materials may 1 also occur as a result of the comparatively high temperatures. 2 3 4 Chemical propellants which have not been converted can locate on the surface 5 of the articles obtained and cause yellowing of the articles. Allergic skin reactions 6 7 may also result upon contact with these articles. 8 Foam articles which have been obtained by means of chemical propellants are 9 not recyclable, or if so only conditionally, since there is the risk that non-ignited 10 propellants can lead to uncontrolled reactions during reuse. 11 12 13 Therefore, physical propellants are preferably used to foam plastics. Physical 14 propellants allow optimum adaptation of the melting temperature to the respectively 15 selected raw material, as a result of which the energy expenditure is reduced, optimum 16 cycle and cooling times are made possible and, in addition, there is no risk that the 17 raw materials could be detrimentally affected as a result of temperatures which are too 18 high. Moreover, inexpensive gases such as CO2, for example, can be used as physical 19 20 propellants. 21 22 Physical propellants do not remain in the finished foam articles, but diffuse out 23 within a comparatively short time. Therefore, these articles are fully recyclable, since 24 there is no need to fear that propellant residues could lead to uncontrolled reactions. 25 26 Various processes are known for the production of articles from foamed plastic 27

with a compact closed external skin and a cellular core cohering with the external skin

or edge zone, also referred to as integral foam or structural foam.

For example, in the reaction injection moulding process (RIM), two reactive components are mixed together which harden and foam in the cavity of a mould under reaction. Because of the quicker cooling at the wall of the mould, the reaction mass solidifies more quickly there than in the interior of the mould, mold and, as a result, the foaming process ceases earlier there than in the mould interior, and a compact sealed external layer is formed.

As determined by the process, the reactive component mixture must be comparatively liquid in order to guarantee complete filling of the mould before the reaction starts. However, this leads to irregularities on the surface of the formed article as a result of spray over and skin formation, which necessitates expensive finishing for high-grade articles, for which a perfect surface is required.

Moreover, for the RIM process the mould must be treated with a separating agent prior to injection, which on the one hand requires more expenditure in processing and can additionally lead to residues on the finished article which must be removed. The relatively long cycle times are also disadvantageous.

#### Since

Because foaming in the RIM process is generally conducted chemically, the articles to be obtained are only conditionally recyclable.

Integral foams made of polyurethane to be used as working material primarily in the automobile industry, e.g. for steering wheel casings or gearshift knobs etc., are preferably produced using the RIM process. However, for this field of application the

articles must not only have as perfect a surface as possible, but also have pleasant skin feel (tactility).

1 2

It has been shown that articles of polyurethane integral foam have only a conditionally acceptable tactility.

It is also known to produce integral foams from thermoplastic urethane or thermoplastic elastomer by means of conventional injection moulding processes. Both chemical and physical propellants can be used in this case. Contrary to the RIM process, which requires special plants, already existing injection moulding plants without expensive refitting can be used for this.

The necessary finishing of the articles obtained is only slight.

DE 196 46 665 A1 describes a process for metering physical propellants, wherein a propellant is added at high pressure to the softened plastic material transported in the consumer, e.g. an extruder or an RIM machine, and the amount of propellant is regulated with a pressure control valve, which keeps the pressure difference constant via a rigid throttle means by regulating the pressure difference in dependence on the flow of propellant. The extrusion processes described here are continuous processes in which the propellant is permanently added.

A process for the production of multilayered articles with a foamed core and a non-expanded thermoplastic external skin is known from DE 1 778 457, wherein a first propellant-free melt and a second melt containing propellant as well as possibly a third propellant-free melt are firstly prepared and injected one after the other into an

appropriate mould, in which case the mould must be maintained at a temperature equal to or higher than the activation temperature of the propellant.

Where physical propellants are used, it is suggested that either the selected temperature of the melt upon leaving the nozzle is so high that, when a mould with constant internal volume is used, the gas formation, and thus the expansion, still occurs below the pressure exerted on the substance in the mould, and when a mould with extendable interior is used, the gas formation, and thus the expansion, occurs by relieving the pressure exerted on the mould interior to expand the mould. There is no mention of the propellant being added directly to the melt flow which flows into the mould, nor of the quantity of propellant apportioned to the melt flow being regulated via the pressure.

An improved process of the aforementioned type is specified in DE 1 948 454, wherein the propellant is injected into the melt flow shortly before entry into the mould and the injection period is continued until the mixture quantity required to form the core has been inserted into the mould. Solvents with a boiling point preferably between 20 and 150°C are specified as propellants, which are to prevent premature expansion under a corresponding pressure. There is likewise no mention here of a pressure regulation of the added quantity of propellant to the melt.

A process for the production of injection moulded articles with foamed core is described in the US pU.S. Patent No. 4,548,776, according to which gaseous of gasgenerated chemical propellant is already added to the melt in the extruder, is thoroughly mixed with this and the already foamed melt is then injected into the mould.

In this case, the addition of propellant occurs via a porous insert at the injection point, a supply valve being provided in the feed pipe. This supply valve can be connected to an automatic control device, via which the pressure of the propellant to be fed is adjusted.

 The object of the present invention disclosure is to provide a process for the production of physically foamed injection moulded articles, with which injection moulded articles with an integral structure, excellent surface characteristics, thus rendering expensive finishing unnecessary, and additionally excellent tactility, can be obtained in a simple manner using conventional injection moulding plants, thus

rendering expensive finishing unnecessary.

# **SUMMARY**

The articles produced according to the invention disclosure are suitable in particular for fields of application which set high quality requirements for surface structure and for which a pleasant sensory feel is of advantage on skin contact. The automobile industry is given as an example, for which handles, knobs such as gearshift knobs, steering wheel casings etc. of the foamed plastics obtained according to the invention disclosure can be used. However, the process according to the invention disclosure is in no way restricted to the production of articles for the automobile industry, but is quite generally suitable for the production of any desired foamed injection moulded articles.

For example, mass-produced articles such as closing means for bottle-like containers, e.g. stoppers or corks, may also be advantageously obtained according to this process. Further examples are balls, spheres, fenders, floats, etc.

1 A further field of use is the production of supporting parts, for example, for the aviation or automobile industry, in particular for parts where strength is relevant. 2 3 4 5 This object is achieved according to the invention disclosure by a process for 6 the production of physically foamed injection moulded articles, wherein firstly in a 7 first stage a propellant-free first melt portion is fed into a cavity (initial filling), in a 8 second stage a physical propellant is added at elevated pressure to the following melt 9 portion (propellant injection phase), wherein metering of the physical propellant 10 occurs at least in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure 11 12 which is exerted on the propellant in the phases between or before or after metered 13 addition, and the expansion of the propellant occurs in the cavity, and possibly in a third stage a propellant-free further melt portion is charged into the cavity. 14 15 16 17 This process also permits the formation of physically foamed injection moulded articles, the foamed core of which is completely or partially enclosed by a 18 19 compact closed external skin, which has been produced without the addition of 20 propellants, the core and the external skin being made of the same material. 21 22 23 The present invention disclosure additionally relates to a device for the metered 24 addition of propellants under elevated pressure to an expandable melt.

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This device can also be advantageously used for the metered addition of compressible propellants.

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1 The propellant-free melt portion firstly fed into the cavity in the first stage 2 forms a compact closed external skin without pores in the finished foamed injection moulded articles. 3 4 5 Any desired fluid which expands upon corresponding pressure relief and 6 foams the melt material in a suitable manner can be used as propellant. Hence, compressible fluids such as gases in liquid or supercritical phase, for example, may 7 8 be used. 9 The use of carbon dioxide is recommended because of its ready availability. 10 11 12 A further preferred propellant is water. 13 The starting material for the melt is not subject to any special restrictions. Any 14 15 desired thermoplastic melt material which is suitable for injection moulding and can 16 be foamed may be used. 17 Examples are thermoplastic materials, but also further thermoplastic melts, 18 such as metallic or ceramic melts, for example. Examples of metallic materials 19 20 include aluminium, magnesium, zinc, tin or even precious metals. 21 The process according to the invention disclosure leads to weight reduction and 22 23 strength increase in comparison to the corresponding compact articles. 24 25 "Pressure regulated" in the sense of the invention disclosure means that in the 26 course of the process the pressure exerted on the propellant is varied for metered addition of the propellant. 27

In this case the pressure exerted on the propellant during the propellant injection phase is greater than the pressure exerted on the propellant in the phases between or before or after metered addition. This means in the case of critical or compressible propellants, for example, that the pressure exerted in the intermediate cycle times is lower than the holding pressure of a pressure relief valve or overflow valve. Therefore, according to the invention disclosure, the required proportion of propellant is added to a melt to be foamed at a defined time over a defined period of time under a defined pressure. 

The magnitude of the pressure exerted on the propellant during the metered addition is determined in particular in dependence on the required quantity of propellant, the type of article to be produced as well as the selected process parameters.

The present invention disclosure is explained in more detail below with reference to the figures on the basis of a preferred embodiment by the example of the addition of a compressible fluid. It goes without saying that the following explanation may also be applied in principle to non-compressible fluids such as water, for example.

# Figures 1a-1d

## BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and other objects and advantages of the embodiments described herein will become apparent with reference to the following detailed description when taken in conjunction with the accompanying drawings in which:

	FIGS, 1A-1D	show the individual stages of the process
		-according to the invention disclosure for the production
		of physically foamed injection moulded
articl	es;	
Figu	<del>re</del>	
	FIG. 2	schematically shows a device for executing the
		─reprocess according to the invention;
Figu	redisclosure;	
	FIG. 3	−is a graph showing the pressure curve during
	- 1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1	- execution of the process;
Figu	re and	
	FIG. 4	shows a variant of FigureFIG. 1 with direct
-	-18	—jintroduction of the propellant into the cavity.
	DET	ALED DESCRIPTION OF THE DRAWINGS
	As Figure II	a lake shows, the cavity 1 of any injection moulding plant is
parti	ally initially fille	ed in a first stage firstly with a compact propellant-free melt 6.
In th	is case, thea fee	d pipe 3 for a compressed propellant is closed, for example, by
a va	lve 4 such as a p	ressure relief valve ﷺ (overflow valve).
	After the cav	ity 1 has been filled with a desired quantity of propellant-free
melt	6, the feed pipe	3 for the propellant is opened and the propellant is injected in
com	pressed, preferal	oly liquid, state via the injection point 5. Through contact with
the l	not melt, the liqu	id propellant turns to gas and expands under the lower pressure
in th	ne cavity.	

1 As a general rule the propellant is still liquid and not gaseous at the injection 2 point 5 itself, and therefore one cannot talk of a "gasification point" in a narrower 3 sense. 4 5 The 6 A mixture 7 of gaseous propellant and melt flows into the cavity 1 and causes 7 the cavity 1 to fill completely, in which case the propellant-free melt portion 6 which 8 was used for the initial filling comes to rest in the region of the cavity walls and forms 9 the external skin or edge zone of the injection moulded article to be formed. 10 11 12 The cavity 1 can be ready filled as desired and required up to the maximum 13 filling quantity with melt mixed with propellant or, as shown in FigureFIG. 1dD, 14 propellant-free melt can again be fed to the cavity in a third stage." In this case a 15 foamed article is obtained which has a compact firm external skin right around which 16 is formed by propellant-free melt. 17 18 19 After foaming and hardening, the finished injection moulded article, e.g. made 20 of integral foam, is removed from the cavity and the cavity is immediately available again for the next charge. 21 22 23 24 As shown in Figure FIG. 1dD, injection moulded articles, which have a cellular 25 foamed internal core and a compact firm closed external skin, are obtained with the 26 process according to the invention disclosure. 27 28 Contrary to the known foaming processes, such as those described above, in

which the cavity is filled completely with a melt/propellant mixture, according to the

inventiondisclosure an initial filling with propellant-free melt occurs firstly, as a result of which the formation of a uniform closed compact external skin is effected and articles with excellent surface characteristics can be obtained. It is essential for execution of the process to prevent premature expansion of the propellant held under pressure. This can be achieved by appropriate insulation of the device and/or maintaining a suitable pressure level. The metered addition of the propellant is conducted in a time- and pressure-controlled manner for the process according to the invention disclosure. Control can be carried out via a device which is also the subject of the invention disclosure. As shown in FigureFIG. 2, the propellant stored under pressure in a storage means 11, e.g. a pressure cylinder etc., is fed to a pressure control valve 10, which can be a multi-way valve such as a 3/3- or 2/3-way proportional valve, and should advantageously have a very quick reaction time and precise regulation. During the propellant injection phase, i.e. the phase in which the propellant is added to the melt, in the case of critical propellants, the compressed propellant passes via a pressure relief valve 4 to the injection point 5 and there is added to the melt. In this case, the dimensions of the pipes, connection pieces and also the parts of the technical control system of the process are such that no premature expansion

in volume of the propellant under pressure is possible.

In the case of a sudden increase in volume the aggregate state of the agent can change, i.e. the agent changes into a gas, in which case vaporisgation cold is generated, which would in turn block the pipes as a result of "icing:":

An increase in temperature on the way to the injection point 5 would also lead to a change in the aggregate state. For prevention, insulation of the heat-carrying elements is recommended.

In order to prevent premature expansion, all feed pipes should be as short as possible. Consequently, the pressure control valve 10 is preferably constructed to be as close as possible to the injection point 5. An improvement to the control characteristics of the control valve is also achieved as a result of the thus shortened feed pipe to the injection point 5.

If critical propellants are used, a pressure relief valve or overflow valve 4 is provided before the injection point 5, this valve ensuring that the pressure in the device does not drop below a specific value, preferably p (crit) at the given temperature, at which the transformation of the propellant into gas would take place. If, for example, carbon dioxide is used as propellant, a pressure of at least 60 bar should be maintained at room temperature in order to keep the carbon dioxide in the device upstream in liquid state.

The pressure relief valve 4 ensures that the propellant remains in compressed state even during outage times of the machine, e.g. in the intermediate cycle times before and after or between the propellant injection phases. A full release of pressure

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phase.

only occurs when the machine or control system is switched off. Several pressure 1 2 relief valves with "falling" pressure values may also be provided so that a pressure 3 gradient is formed in front of the injection point 5 in the feed pipe section between the 4 pressure control valve 10 and the pressure relief valve 4. 5 6 7 The graph in FigureFIG. 3 schematically shows the pressure curve for 8 executing the process according to the invention disclosure using the example of 9 compressible propellants. 10 11 12 Outside of the propellant injection phase, as in the intermediate cycle times, 13 it is sufficient to keep the device at a selected pressure, at which the propellant 14 respectively used remains in compressed, preferably liquid, state (section 20). 15 16 During the propellant injection phase (section 22), an elevated pressure is 17 18 introduced in the feed pipes through the pressure control valve 10 so that the opening 19 pressure (holding pressure) of the relief valve 4 is exceeded and the feed pipe section 20 3 up to the injection point 5 is quickly filled with liquid medium. 21 22 23 In this case, the pressure increase is proportional to the desired quantity of propellant to be fed to the melt. After time time as soon as the desired 24 25 quantity of propellant has been added to the melt, the pressure is reduced again to the 26 starting pressure (section 24). 27

In Figure 116. 3, sections 21 and 23 show the pressure build up or reduction

The injection point 5 is preferably configured as a throttle means, e.g. as a defined gap in an injector, a sintered metal injector, or a needle valve. According to the invention disclosure, a controlled closure mechanism is located at the injection point. The quick pressure increase and the resistance through the injector prevent the propellant from transforming into gas, while the agent flows on from the pressure control valve 10.

The above measures ensure that the transformation of the agent into gas only occurs upon exit from the injector and when in contact with the hot melt, and that the

inflowing melt is foamed.

The controlled closure mechanism can be omitted if a pressure relief valve is provided.

After the propellant injection phase has ended, i.e. after the desired quantity of propellant has been added to the melt, the pressure in the feed pipe to the injection point 5 is reduced so that no propellant can flow on. However, in the pipe up to the pressure relief valve 4 the starting pressure remains in order to keep the agent in compressed or liquid state for the next cycle. A virtually pressure-free and thus gaseous state prevails only in the short feed pipe section from the pressure relief valve 4 to the injection point 5 until the next cycle.

It goes without saying that this part of the plant may also be kept under pressure if required by the provision of a suitable closure mechanism which opens

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again at the beginning of the propellant injection phase as a result of the increasing 1 2 pressure level. 3 4 The pressure control via the pressure control valve can occur automatically by 5 providing pressure measurement points 12, 13, for example, in front of and behind the 6 7 pressure control valve. 8 9 If carbon dioxide is used as propellant, for example, the plant is preferably 10 held at an operating pressure of at least 60 bar at room temperature, so that the CO<sub>2</sub> 11 12 also remains in compressed state during the periods between the propellant injection phases. At the beginning of the propellant injection phase, a desired working pressure 13 of about 200 bar, for example, is built up (section 21) in order to assure an adequate 14 flow of propellant to the melt. After the propellant injection phase 22 has ended, the 15 pressure is reduced again to the desired operating pressure. 16 17 The injection point 5 is preferably located in the feeder pipe 3 close to the 18 spray point xix." According to a further embodiment, as is shown in FigureFIG. 4, 19 20 the propellant can be added directly to the melt in the cavity. In this case, the injection point 5 is located directly at the cavity. 21 22 23 In addition, the build up of a counterpressure can be provided in the cavity 1, 24 such as is also used in conventional injection moulding processes in the so-called gas 25 counterpressure process. 26 27

Very short cycle times can be obtained with the process according to the

invention disclosure. Hence, the process according to the invention disclosure is also

very well suited to the production of mass-produced articles. The short cycle times are supported by the vaporiszation cold resulting upon the transformation of the propellant into gas, and this causes a reduction in the cooling time, and thus also the cycle time.

Should there still be propellant residues present in the pore structure in the core of the article after demoulding, these slowly diffuse out of the article without detriment to its usability or recyclability.

Excellent dimensional stability of the article is achieved as a result of its closed firm external skin. In addition, foamed injection moulded articles which have a homogeneous uniform external skin and excellent tactility can be obtained with the process according to the invention disclosure.

The foamed injection moulded articles obtained have an excellent surface quality and do not require any further finishing. It is also of advantage that the cavity does not need to be treated with a separating agent.

The process according to the invention disclosure for the pressure-controlled metered addition of physical propellants to an expandable melt can be conducted advantageously with a device comprising a storage means 11, in which the propellant is stored under pressure, a pressure control valve 10 for regulating the propellant pressure and an injection point 5, which is preferably configured as a throttle means, at which the propellant under pressure is added to the melt, wherein the injection point 5 includes a controlled closure mechanism, and in the case of critical propellants at

least one pressure relief valve 4 is provided which is positioned downstream of the pressure control valve 10.

Although the above-described process and the device for the pressure-controlled metered addition of propellants under high pressure can be advantageously used for the production of physically foamed injection moulded articles, they are, of course, also suitable for other processes in which propellants are added under high pressure to melts to be expanded.

1.	List of Refer	rence Numbers
2		
3		
4	1	<del>- cavity</del>
5	2	<del>melt feed</del>
6	3	propellant feed pipe
7	4	<del>pressure relief valve</del>
8	5	injection point
9	6	<del>propellant-free melt</del>
10	7	melt with added propellant
11	8	injection of plastic material
12	9	mould comprising two halves
13	10	pressure control valve
14	11	<del>propellant storage means</del>
15		
16	×	spray point
17		
18	Section 20	pressure during the intermediate eyele
19	times	
20	Section 21	pressure build up phase
21	Section 22	propellant injection phase
22	Section 23	pressure reduction phase
23		
24		
25		
26		
27		
28		
29		

1	Claims:
2	
3	1. Process for the production of
1	What is claimed is

given temperature.

#### PROCESS FOR THE PRODUCTION OF PHYSICALLY FOAMED 1 INJECTION MOLDED ARTICLES 2 3 ABSTRACT A process for producing physically foamed injection moulded articles, 4 5 wherein in a first stage is provided. The process involves feeding a propellant-free 6 first melt portion (6) is fed into a cavity (1) (initial filling), in a second 7 stagethermoplastic melt into a cavity followed by delivering a physical propellant is added at elevated pressure to the following melt portion (propellant injection 8 9 phase), and possibly in a third stage a propellant-free further melt portion is chargeddirectly into the cavity (1), the production of the injection moulded articles 10 11 occurring in the cavity, 12 characterised in that metering of the physical propellant in the second stage occurs 13 in a pressure regulated manner, wherein the pressure which isor directly into the melt flowing into the cavity. The pressure exerted on the propellant during the 14 propellant injection phase stage is greater than the pressure which is that exerted on 15 16 the propellant in the phases between or before or after metered addition, and the 17 expansion of the propellant occurs in the cavity (1). 18 Process according to Claim 1, characterised in that 19 20 the propellant is a compressible fluid. 21 3. Process according to Claim 1 or 2, characterised in that 22 the propellant is kept under pressure in the intermediate eyele times before and 23 after the propellant injection phase, or is present in a compressed state. 24 25 Process according to Claim 3, characterised in that 26 in the intermediate eyele times the propellant is held a 27 pressure of at least p (erit) of the propellant at the 28

1	5. Process according to one of the preceding claims,
2	eharacterised in that the pressure exerted on the
3	propellant is controlled via a pressure control valve (10).
4	
5	6. Process according to Claim 5, characterised in that
6	the pressure control valve (10) is a multi-way valve.
7	
8	7. Process according to Claim 6, characterised in that
9	a 3/3-way proportional valve or a 2/3-way proportional valve is used as multi-way
10	<del>valve.</del>
11	
12	8. Process according to one of the preceding claims, characterised in that the
13	pressure control in the case of critical propellants additionally occurs via at least
14	one pressure relief valve (4) which is connected downstream of the pressure
15	control valve (10).
16	
17	9. Process according to Claim 8, characterised in that
18	the holding pressure of at least one of the pressure relief valves (4) is equal to or
19	higher than the pressure at which a critical propellant is held in the intermediate
20	eyele times.
21	
22	10. Process according to one of the preceding claims, characterised in that the
23	pressure preset by the pressure control valve (10) is regulated via one or more
24	pressure relief valves (4) to the injection pressure at which the propellant is added
25	to the melt via an injection point (5).
26	
27	11. Process according to one of the preceding claims, characterised in that the
28	injection point (5) is configured as a throttle means.
29	

1	12. Process according to Claim 11, characterised in that
2	the injection point (5) is in the form of a defined gap in an injector or of an injecto
3	with a sinter metal.
4	
5	13. Process according to one of Claims 11 or 12,
6	characterised in that the injection point (5) is configured as a controlled closure
7	mechanism.
8	
9	14. Process according to Claim 1 or one of the preceding Claims 3 to 13,
10	eharacterised in that water is used as propellant.
11	
12	15. Process according to one of the preceding Claims 1 to 13, characterised in
13	that a gas or gas mixture is used as propellant.
14	
15	16. Process according to Claims 15, characterised in that
16	earbon dioxide is used as propellant.
17	
18	17. Process according to Claims 16, characterised in that
19	the earbon dioxide is held in the intermediate eyele times at a pressure of at least
20	60 bar (= p (crit) CO <sub>2</sub> at room temperature).
21	
22	18. Process according to one of the preceding claims, characterised in that for
23	the propellant injection phase the propellant is brought to a pressure of over 60 ba
24	via the pressure control valve (10).
25	
26	19. Process according to one of the preceding claims, characterised in that a
27	counterpressure is generated in the cavity (1).
28	
29	20 Process according to one of the preceding claims.

1	characterised in that the physically foamed injection moulded article is selected
2	from a handle, a knob, a gearshift knob, a steering wheel easing, a ball, a sphere, a
3	fender, a float and a closing means for bottle-like containers.
4	
5	21. Device for the metered addition of physical propellants to a foamable melt
6	wherein the device comprises a storage means (11), in which the propellant is
7	stored under pressure, a pressure control valve (10) for regulating the propellant
8	pressure, and an injection point (5), which is configured as a throttle means, at
9	which the propellant under pressure is fed to the melt,
10	characterised in that a controlled closure mechanism is provided at the injection
l 1	point (5).
12	
13	22. Device for the metered addition of physical propellants according to Clain
14	21, characterised in that instead of the controlled closure mechanism or in addition
15	to the controlled closure mechanism, at least one pressure relief valve (4) is
16	<del>provided.</del>
17	
18	during the holding phase.

## PROCESS FOR THE PRODUCTION OF PHYSICALLY FOAMED

## **INJECTION MOLDED ARTICLES**

#### BACKGROUND

### Technical Field

The present disclosure relates to a process for the production of physically foamed injection molded articles and, in particular, to a process for the production of physically foamed injection molded articles with an internal foam structure and a compact closed-pore external skin of the same material as the base body.

## Related Art

The production of foamed plastics, for example, is achieved with the aid of socalled propellants, which expand a plastic, generally thermally softened plastic mass in the desired manner. In this case, the propellants are either generated in situ via chemical reaction of the components (chemical propellants), or compressed fluids, e.g.  $N_2$ ,  $CO_2$ , are added under pressure to the starting material, in which case a foaming process of the plastic mass caused by the propellant is initiated upon the subsequent drop in pressure of the component mixture to normal pressure.

However, chemical propellants have a series of disadvantages. For instance, for use in foam injection molding higher temperatures than are actually necessary for softening starting materials may have to be selected in order to reach the ignition point of the propellants, since the temperature at which the reaction of the components generating propellant starts is generally very high. Because of the high temperatures a higher expenditure of energy is necessary during melting of the raw materials. In addition, the cycle or cooling times are increased and a higher cooling power of the cooling plants is necessary. In some circumstances, damage to the raw materials may also occur as a result of the comparatively high temperatures.

Chemical propellants which have not been converted can locate on the surface of the articles obtained and cause yellowing of the articles. Allergic skin reactions may also result upon contact with these articles.

Foam articles which have been obtained by means of chemical propellants are not recyclable, or if so only conditionally, since there is the risk that non-ignited propellants can lead to uncontrolled reactions during reuse.

Therefore, physical propellants are preferably used to foam plastics. Physical propellants allow optimum adaptation of the melting temperature to the respectively selected raw material, as a result of which the energy expenditure is reduced, optimum cycle and cooling times are made possible and, in addition, there is no risk that the raw materials could be detrimentally affected as a result of temperatures which are too high. Moreover, inexpensive gases such as CO<sub>2</sub>, for example, can be used as physical propellants.

Physical propellants do not remain in the finished foam articles, but diffuse out within a comparatively short time. Therefore, these articles are fully recyclable, since there is no need to fear that propellant residues could lead to uncontrolled reactions.

Various processes are known for the production of articles from foamed plastic with a compact closed external skin and a cellular core cohering with the external skin or edge zone, also referred to as integral foam or structural foam.

For example, in the reaction injection molding process (RIM), two reactive components are mixed together which harden and foam in the cavity of a mold under reaction. Because of the quicker cooling at the wall of the mold, the reaction mass solidifies more quickly there than in the interior of the mold and, as a result, the foaming process ceases earlier there than in the mold interior, and a compact sealed external layer is formed.

As determined by the process, the reactive component mixture must be comparatively liquid in order to guarantee complete filling of the mold before the reaction starts. However, this leads to irregularities on the surface of the formed

~ 4

article as a result of spray over and skin formation, which necessitates expensive finishing for high-grade articles, for which a perfect surface is required.

Moreover, for the RIM process the mold must be treated with a separating agent prior to injection, which on the one hand requires more expenditure in processing and can additionally lead to residues on the finished article which must be removed. The relatively long cycle times are also disadvantageous.

Because foaming in the RIM process is generally conducted chemically, the articles to be obtained are only conditionally recyclable.

Integral foams made of polyurethane to be used as working material primarily in the automobile industry, e.g. for steering wheel casings or gearshift knobs etc., are preferably produced using the RIM process. However, for this field of application the articles must not only have as perfect a surface as possible, but also have pleasant skin feel (tactility).

It has been shown that articles of polyurethane integral foam have only a conditionally acceptable tactility.

It is also known to produce integral foams from thermoplastic urethane or thermoplastic elastomer by means of conventional injection molding processes. Both chemical and physical propellants can be used in this case. Contrary to the RIM process, which requires special plants, already existing injection molding plants without expensive refitting can be used for this.

The necessary finishing of the articles obtained is only slight.

DE 196 46 665 A1 describes a process for metering physical propellants, wherein a propellant is added at high pressure to the softened plastic material transported in the consumer, e.g. an extruder or an RIM machine, and the amount of propellant is regulated with a pressure control valve, which keeps the pressure difference constant via a rigid throttle means by regulating the pressure difference in dependence on the flow of propellant. The extrusion processes described here are continuous processes in which the propellant is permanently added.

A process for the production of multilayered articles with a foamed core and a non-expanded thermoplastic external skin is known from DE 1 778 457, wherein a first propellant-free melt and a second melt containing propellant as well as possibly a third propellant-free melt are firstly prepared and injected one after the other into an appropriate mold, in which case the mold must be maintained at a temperature equal to or higher than the activation temperature of the propellant.

Where physical propellants are used, it is suggested that either the selected temperature of the melt upon leaving the nozzle is so high that, when a mold with constant internal volume is used, the gas formation, and thus the expansion, still occurs below the pressure exerted on the substance in the mold, and when a mold with extendable interior is used, the gas formation, and thus the expansion, occurs by relieving the pressure exerted on the mold interior to expand the mold. There is no mention of the propellant being added directly to the melt flow which flows into the mold, nor of the quantity of propellant apportioned to the melt flow being regulated via the pressure.

An improved process of the aforementioned type is specified in DE 1 948 454, wherein the propellant is injected into the melt flow shortly before entry into the mold and the injection period is continued until the mixture quantity required to form the core has been inserted into the mold. Solvents with a boiling point preferably between 20 and 150°C are specified as propellants, which are to prevent premature expansion under a corresponding pressure. There is likewise no mention here of a pressure regulation of the added quantity of propellant to the melt.

A process for the production of injection molded articles with foamed core is described in U.S. Patent No. 4,548,776, according to which gaseous of gas-generated chemical propellant is already added to the melt in the extruder, is thoroughly mixed with this and the already foamed melt is then injected into the mold.

In this case, the addition of propellant occurs via a porous insert at the injection point, a supply valve being provided in the feed pipe. This supply valve can

be connected to an automatic control device, via which the pressure of the propellant to be fed is adjusted.

The object of the present disclosure is to provide a process for the production of physically foamed injection molded articles, with which injection molded articles with an integral structure, excellent surface characteristics, and excellent tactility, can be obtained in a simple manner using conventional injection molding plants, thus rendering expensive finishing unnecessary.

#### **SUMMARY**

The articles produced according to the disclosure are suitable in particular for fields of application which set high quality requirements for surface structure and for which a pleasant sensory feel is of advantage on skin contact. The automobile industry is given as an example, for which handles, knobs such as gearshift knobs, steering wheel casings etc. of the foamed plastics obtained according to the disclosure can be used. However, the process according to the disclosure is in no way restricted to the production of articles for the automobile industry, but is quite generally suitable for the production of any desired foamed injection molded articles.

For example, mass-produced articles such as closing means for bottle-like containers, e.g. stoppers or corks, may also be advantageously obtained according to this process. Further examples are balls, spheres, fenders, floats, etc.

A further field of use is the production of supporting parts, for example, for the aviation or automobile industry, in particular for parts where strength is relevant.

This object is achieved according to the disclosure by a process for the production of physically foamed injection molded articles, wherein firstly in a first stage a propellant-free first melt portion is fed into a cavity (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), wherein metering of the physical propellant occurs at least in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure which is

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exerted on the propellant in the phases between or before or after metered addition, 1 and the expansion of the propellant occurs in the cavity, and possibly in a third stage 2 3 a propellant-free further melt portion is charged into the cavity. This process also permits the formation of physically foamed injection molded 4 articles, the foamed core of which is completely or partially enclosed by a compact 5 6 closed external skin, which has been produced without the addition of propellants, the 7 core and the external skin being made of the same material. The present disclosure additionally relates to a device for the metered addition 8 9 of propellants under elevated pressure to an expandable melt. 10 This device can also be advantageously used for the metered addition of 11 compressible propellants. The propellant-free melt portion firstly fed into the cavity in the first stage 12 forms a compact closed external skin without pores in the finished foamed injection 13 14 molded articles. 15 Any desired fluid which expands upon corresponding pressure relief and foams the melt material in a suitable manner can be used as propellant. Hence, 16 compressible fluids such as gases in liquid or supercritical phase, for example, may 17 18 be used. The use of carbon dioxide is recommended because of its ready availability. 19 20 A further preferred propellant is water. 21 The starting material for the melt is not subject to any special restrictions. Any desired thermoplastic melt material which is suitable for injection molding and can 22 23 be foamed may be used. 24 Examples are thermoplastic materials, but also further thermoplastic melts, such as metallic or ceramic melts, for example. Examples of metallic materials 25

The process according to the disclosure leads to weight reduction and strength increase in comparison to the corresponding compact articles.

include aluminum, magnesium, zinc, tin or even precious metals.

"Pressure regulated" in the sense of the disclosure means that in the course of the process the pressure exerted on the propellant is varied for metered addition of the propellant. In this case the pressure exerted on the propellant during the propellant injection phase is greater than the pressure exerted on the propellant in the phases between or before or after metered addition. This means in the case of critical or compressible propellants, for example, that the pressure exerted in the intermediate cycle times is lower than the holding pressure of a pressure relief valve or overflow valve.

Therefore, according to the disclosure, the required proportion of propellant is added to a melt to be foamed at a defined time over a defined period of time under a defined pressure.

The magnitude of the pressure exerted on the propellant during the metered addition is determined in particular in dependence on the required quantity of propellant, the type of article to be produced as well as the selected process parameters.

The present disclosure is explained in more detail below with reference to the figures on the basis of a preferred embodiment by the example of the addition of a compressible fluid. It goes without saying that the following explanation may also be applied in principle to non-compressible fluids such as water, for example.

## BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and other objects and advantages of the embodiments described herein will become apparent with reference to the following detailed description when taken in conjunction with the accompanying drawings in which:

FIGS. 1A-1D show the individual stages of the process according to the disclosure for the production of physically foamed injection molded articles;

FIG. 2 schematically shows a device for executing the process according to the disclosure;

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1	FIG. 3 is a graph showing the pressure curve during execution of the process;
2	and
3	FIG. 4 shows a variant of FIG. 1 with direct introduction of the propellant into
4	the cavity.
5	
6	DETAILED DESCRIPTION OF THE DRAWINGS
7	As FIG. 1A shows, the cavity 1 of any injection molding plant is partially
8	initially filled in a first stage firstly with a compact propellant-free melt 6. In this
9	case, a feed pipe 3 for a compressed propellant is closed, for example, by a valve 4
10	such as a pressure relief valve 4 (overflow valve).
11	After the cavity 1 has been filled with a desired quantity of propellant-free
12	melt 6, the feed pipe 3 for the propellant is opened and the propellant is injected in
13	compressed, preferably liquid, state via the injection point 5. Through contact with
14	the hot melt, the liquid propellant turns to gas and expands under the lower pressure
15	in the cavity.
16	As a general rule the propellant is still liquid and not gaseous at the injection
17	point 5 itself, and therefore one cannot talk of a "gasification point" in a narrower
18	sense.
19	A mixture 7 of gaseous propellant and melt flows into the cavity 1 and causes
20	the cavity 1 to fill completely, in which case the propellant-free melt portion 6 which
21	was used for the initial filling comes to rest in the region of the cavity walls and forms
22	the external skin or edge zone of the injection molded article to be formed.
23	The cavity 1 can be ready filled as desired and required up to the maximum

maximum filling quantity with melt mixed with propellant or, as shown in FIG. 1D, propellantfree melt can again be fed to the cavity in a third stage. In this case a foamed article is obtained which has a compact firm external skin right around which is formed by propellant-free melt.

After foaming and hardening, the finished injection molded article, e.g. made of integral foam, is removed from the cavity and the cavity is immediately available again for the next charge.

As shown in FIG. 1D, injection molded articles, which have a cellular foamed internal core and a compact firm closed external skin, are obtained with the process according to the disclosure.

Contrary to the known foaming processes, such as those described above, in which the cavity is filled completely with a melt/propellant mixture, according to the disclosure an initial filling with propellant-free melt occurs firstly, as a result of which the formation of a uniform closed compact external skin is effected and articles with excellent surface characteristics can be obtained.

It is essential for execution of the process to prevent premature expansion of the propellant held under pressure. This can be achieved by appropriate insulation of the device and/or maintaining a suitable pressure level.

The metered addition of the propellant is conducted in a time- and pressurecontrolled manner for the process according to the disclosure. Control can be carried out via a device which is also the subject of the disclosure.

As shown in FIG. 2, the propellant stored under pressure in a storage means 11, e.g. a pressure cylinder etc., is fed to a pressure control valve 10, which can be a multi-way valve such as a 3/3- or 2/3-way proportional valve, and should advantageously have a very quick reaction time and precise regulation.

During the propellant injection phase, i.e. the phase in which the propellant is added to the melt, in the case of critical propellants, the compressed propellant passes via a pressure relief valve 4 to the injection point 5 and there is added to the melt.

In this case, the dimensions of the pipes, connection pieces and also the parts of the technical control system of the process are such that no premature expansion in volume of the propellant under pressure is possible.

In the case of a sudden increase in volume the aggregate state of the agent can change, i.e. the agent changes into a gas, in which case vaporization cold is generated, which would in turn block the pipes as a result of "icing."

An increase in temperature on the way to the injection point 5 would also lead to a change in the aggregate state. For prevention, insulation of the heat-carrying elements is recommended.

In order to prevent premature expansion, all feed pipes should be as short as possible. Consequently, the pressure control valve 10 is preferably constructed to be as close as possible to the injection point 5. An improvement to the control characteristics of the control valve is also achieved as a result of the thus shortened feed pipe to the injection point 5.

If critical propellants are used, a pressure relief valve or overflow valve 4 is provided before the injection point 5, this valve ensuring that the pressure in the device does not drop below a specific value, preferably p (crit) at the given temperature, at which the transformation of the propellant into gas would take place. If, for example, carbon dioxide is used as propellant, a pressure of at least 60 bar should be maintained at room temperature in order to keep the carbon dioxide in the device upstream in liquid state.

The pressure relief valve 4 ensures that the propellant remains in compressed state even during outage times of the machine, e.g. in the intermediate cycle times before and after or between the propellant injection phases. A full release of pressure only occurs when the machine or control system is switched off. Several pressure relief valves with "falling" pressure values may also be provided so that a pressure gradient is formed in front of the injection point 5 in the feed pipe section between the pressure control valve 10 and the pressure relief valve 4.

The graph in FIG. 3 schematically shows the pressure curve for executing the process according to the disclosure using the example of compressible propellants.

Outside of the propellant injection phase, as in the intermediate cycle times, it is sufficient to keep the device at a selected pressure, at which the propellant respectively used remains in compressed, preferably liquid, state (section 20).

During the propellant injection phase (section 22), an elevated pressure is introduced in the feed pipes through the pressure control valve 10 so that the opening pressure (holding pressure) of the relief valve 4 is exceeded and the feed pipe section 3 up to the injection point 5 is quickly filled with liquid medium.

In this case, the pressure increase is proportional to the desired quantity of propellant to be fed to the melt. After time "t" expires, as soon as the desired quantity of propellant has been added to the melt, the pressure is reduced again to the starting pressure (section 24).

In FIG. 3, sections 21 and 23 show the pressure build up or reduction phase.

The injection point 5 is preferably configured as a throttle means, e.g. as a defined gap in an injector, a sintered metal injector, or a needle valve. According to the disclosure, a controlled closure mechanism is located at the injection point. The quick pressure increase and the resistance through the injector prevent the propellant from transforming into gas, while the agent flows on from the pressure control valve 10.

The above measures ensure that the transformation of the agent into gas only occurs upon exit from the injector and when in contact with the hot melt, and that the inflowing melt is foamed.

The controlled closure mechanism can be omitted if a pressure relief valve is provided.

After the propellant injection phase has ended, i.e. after the desired quantity of propellant has been added to the melt, the pressure in the feed pipe to the injection point 5 is reduced so that no propellant can flow on. However, in the pipe up to the pressure relief valve 4 the starting pressure remains in order to keep the agent in compressed or liquid state for the next cycle. A virtually pressure-free and thus

gaseous state prevails only in the short feed pipe section from the pressure relief valve 4 to the injection point 5 until the next cycle.

It goes without saying that this part of the plant may also be kept under pressure if required by the provision of a suitable closure mechanism which opens again at the beginning of the propellant injection phase as a result of the increasing pressure level.

The pressure control via the pressure control valve can occur automatically by providing pressure measurement points 12, 13, for example, in front of and behind the pressure control valve.

If carbon dioxide is used as propellant, for example, the plant is preferably held at an operating pressure of at least 60 bar at room temperature, so that the CO<sub>2</sub> also remains in compressed state during the periods between the propellant injection phases. At the beginning of the propellant injection phase, a desired working pressure of about 200 bar, for example, is built up (section 21) in order to assure an adequate flow of propellant to the melt. After the propellant injection phase 22 has ended, the pressure is reduced again to the desired operating pressure.

The injection point 5 is preferably located in the feeder pipe 3 close to the spray point "x." According to a further embodiment, as is shown in FIG. 4, the propellant can be added directly to the melt in the cavity. In this case, the injection point 5 is located directly at the cavity.

In addition, the build up of a counterpressure can be provided in the cavity 1, such as is also used in conventional injection molding processes in the so-called gas counterpressure process.

Very short cycle times can be obtained with the process according to the disclosure. Hence, the process according to the disclosure is also very well suited to the production of mass-produced articles. The short cycle times are supported by the vaporization cold resulting upon the transformation of the propellant into gas, and this causes a reduction in the cooling time, and thus also the cycle time.

Should there still be propellant residues present in the pore structure in the core of the article after demolding, these slowly diffuse out of the article without detriment to its usability or recyclability.

Excellent dimensional stability of the article is achieved as a result of its closed firm external skin. In addition, foamed injection molded articles which have a homogeneous uniform external skin and excellent tactility can be obtained with the process according to the disclosure.

The foamed injection molded articles obtained have an excellent surface quality and do not require any further finishing. It is also of advantage that the cavity does not need to be treated with a separating agent.

The process according to the disclosure for the pressure-controlled metered addition of physical propellants to an expandable melt can be conducted advantageously with a device comprising a storage means 11, in which the propellant is stored under pressure, a pressure control valve 10 for regulating the propellant pressure and an injection point 5, which is preferably configured as a throttle means, at which the propellant under pressure is added to the melt, wherein the injection point 5 includes a controlled closure mechanism, and in the case of critical propellants at least one pressure relief valve 4 is provided which is positioned downstream of the pressure control valve 10.

Although the above-described process and the device for the pressurecontrolled metered addition of propellants under high pressure can be advantageously used for the production of physically foamed injection molded articles, they are, of course, also suitable for other processes in which propellants are added under high pressure to melts to be expanded.

What is claimed is:

09/936756 = NGLISH TRANSLATION OF APPLICITATION SEPTIMENTAL

# Process for the Production of Physically Foamed Injection Moulded Articles

The present invention relates to a process for the production of physically foamed injection moulded articles, in particular injection moulded articles with an internal foam structure and a compact closed-pore external skin of the same material as the base body.

The production of foamed plastics, for example, is achieved with the aid of so-called propellants, which expand a plastic, generally thermally softened plastic mass in the desired manner. In this case, the propellants are either generated in situ via chemical reaction of the components (chemical propellants), or compressed fluids, e.g.  $N_2$ ,  $CO_2$ , are added under pressure to the starting material, in which case a foaming process of the plastic mass caused by the propellant is initiated upon the subsequent drop in pressure of the component mixture to normal pressure.

However, chemical propellants have a series of disadvantages. For instance, for use in foam injection moulding higher temperatures than are actually necessary for softening starting materials may have to be selected in order to reach the ignition point of the propellants, since the temperature at which the reaction of the components generating propellant starts is generally very high. Because of the high temperatures a higher expenditure of energy is necessary during melting of the raw materials. In addition, the cycle or cooling times are increased and a higher cooling power of the cooling plants is necessary. In some circumstances, damage to the raw materials may also occur as a result of the comparatively high temperatures.

Chemical propellants which have not been converted can locate on the surface of the articles obtained and cause yellowing of the articles. Allergic skin reactions may also result upon contact with these articles.

Foam articles which have been obtained by means of chemical propellants are not recyclable, or if so only conditionally, since there is the risk that non-ignited propellants can lead to uncontrolled reactions during reuse.

Therefore, physical propellants are preferably used to foam plastics. Physical propellants allow optimum adaptation of the melting temperature to the respectively selected raw material, as a result of which the energy expenditure is reduced, optimum cycle and cooling times are made possible and in addition there is no risk that the raw materials could be detrimentally affected as a result of temperatures which are too high. Moreover, inexpensive gases such as CO<sub>2</sub>, for example, can be used as physical propellants.

Physical propellants do not remain in the finished foam articles, but diffuse out within a comparatively short time. Therefore, these articles are fully recyclable, since there is no need to fear that propellant residues could lead to uncontrolled reactions.

Various processes are known for the production of articles from foamed plastic with a compact closed external skin and a cellular core cohering with the external skin or edge zone, also referred to as integral foam or structural foam.

For example, in the reaction injection moulding process (RIM), two reactive components are mixed together which harden and foam in the cavity of a mould under reaction. Because of the quicker cooling at the wall of the mould, the reaction mass solidifies more quickly there than in the interior of the mould, and as a result the foaming process ceases earlier there than in the mould interior, and a compact sealed external layer is formed.

As determined by the process, the reactive component mixture must be comparatively liquid in order to guarantee complete filling of the mould before the reaction starts. However, this leads to irregularities on the surface of the formed article as a result of spray over and skin formation, which necessitates expensive finishing for high-grade articles, for which a perfect surface is required.

Moreover, for the RIM process the mould must be treated with a separating agent prior to injection, which on the one hand requires more expenditure in processing and can additionally lead to residues on the finished article which must be removed. The relatively long cycle times are also disadvantageous.

Since foaming in the RIM process is generally conducted chemically, the articles to be obtained are only conditionally recyclable.

Integral foams made of polyurethane to be used as working material primarily in the automobile industry, e.g. for steering wheel casings or gearshift knobs etc., are preferably produced using the RIM process. However, for this field of application the articles must not only have as perfect a surface as possible, but also have pleasant skin feel (tactility).

It has been shown that articles of polyurethane integral foam have only a conditionally acceptable tactility.

It is also known to produce integral foams from thermoplastic urethane or thermoplastic elastomer by means of conventional injection moulding processes. Both chemical and physical propellants can be used in this case. Contrary to the RIM process, which requires special plants, already existing injection moulding plants without expensive refitting can be used for this.

The necessary finishing of the articles obtained is only slight.

DE 196 46 665 Al describes a process for metering physical propellants, wherein a propellant is added at high pressure to the softened plastic material transported in the consumer, e.g. an extruder or an RIM machine, and the amount of propellant is regulated with a pressure control valve, which keeps the pressure difference constant via a rigid throttle means by regulating the pressure difference in dependence on the flow of propellant. The extrusion processes described here are continuous processes in which the propellant is permanently added.

A process for the production of multilayered articles with a foamed core and a non-expanded thermoplastic external skin is known from DE 1 778 457, wherein a first propellant-free melt and a second melt containing propellant as well as possibly a third propellant-free melt are firstly prepared and injected one after the other into an appropriate mould, in which case the mould must be maintained at a temperature equal to or higher than the activation temperature of the propellant.

Where physical propellants are used, it is suggested that either the selected temperature of the melt upon leaving the nozzle is so high that, when a mould with constant internal volume is used, the gas formation, and thus the expansion, still occurs below the pressure exerted on the substance in the mould, and when a mould with extendable interior is used, the gas formation, and thus the expansion, occurs by relieving the pressure exerted on the mould interior to expand the mould. There is no mention of the propellant being added directly to the melt flow which flows into the mould, nor of the quantity of propellant apportioned to the melt flow being regulated via the pressure.

An improved process of the aforementioned type is specified in DE 1 948 454, wherein the propellant is injected into the melt

flow shortly before entry into the mould and the injection period is continued until the mixture quantity required to form the core has been inserted into the mould. Solvents with a boiling point preferably between 20 and 150°C are specified as propellants, which are to prevent premature expansion under a corresponding pressure. There is likewise no mention here of a pressure regulation of the added quantity of propellant to the melt.

The object of the present invention is to provide a process for the production of physically foamed injection moulded articles, with which injection moulded articles with an integral structure, excellent surface characteristics, thus rendering expensive finishing unnecessary, and additionally excellent tactility, can be obtained in a simple manner using conventional injection moulding plants.

The articles produced according to the invention are suitable in particular for fields of application which set high quality requirements for surface structure and for which a pleasant sensory feel is of advantage on skin contact. The automobile industry is given as an example, for which handles, knobs such as gearshift knobs, steering wheel casings etc. of the foamed plastics obtained according to the invention can be used. However, the process according to the invention is in no way restricted to the production of articles for the automobile industry, but is quite generally suitable for the production of any desired foamed injection moulded articles.

For example, mass-produced articles such as closing means for bottle-like containers, e.g. stoppers or corks, may also be advantageously obtained according to this process. Further examples are balls, spheres, fenders, floats etc.

A further field of use is the production of supporting parts, for example, for the aviation or automobile industry, in particular for parts where strength is relevant. This object is achieved according to the invention by a process for the production of physically foamed injection moulded articles, wherein firstly in a first stage a propellant-free first melt portion is fed into a cavity (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), wherein metering of the physical propellant occurs at least in a pressure regulated manner, and possibly in a third stage a propellant-free further melt portion is charged into the cavity.

The present invention also relates to physically foamed injection moulded articles, the foamed core of which is completely or partially enclosed by a compact closed external skin, which has been produced without the addition of propellants, the core and the external skin being made of the same material.

The present invention additionally relates to a device for the metered addition of propellants under elevated pressure to an expandable melt.

This device can also be advantageously used for the metered addition of compressible propellants.

The propellant-free melt portion firstly fed into the cavity in the first stage forms a compact closed external skin without pores in the finished foamed injection moulded articles.

Any desired fluid which expands upon corresponding pressure relief and foams the melt material in a suitable manner can be used as propellant. Hence, compressible fluids such as gases in liquid or supercritical phase, for example, may be used.

The use of carbon dioxide is recommended because of its ready availability.

A further preferred propellant is water.

The starting material for the melt is not subject to any special restrictions. Any desired thermoplastic melt material which is suitable for injection moulding and can be foamed may be used.

Examples are thermoplastic materials, but also further thermoplastic melts, such as metallic or ceramic melts, for example. Examples of metallic materials include aluminium, magnesium, zinc, tin or even precious metals.

The process according to the invention leads to weight reduction and strength increase in comparison to the corresponding compact articles.

"Pressure regulated" in the sense of the invention means that in the course of the process the pressure exerted on the propellant is varied for metered addition of the propellant. In this case the pressure exerted on the propellant during the propellant injection phase is greater than the pressure exerted on the propellant in the phases between or before or after metered addition. This means in the case of critical or compressible propellants, for example, that the pressure exerted in the intermediate cycle times is lower than the holding pressure of a pressure relief valve or overflow valve.

Therefore, according to the invention the required proportion of propellant is added to a melt to be foamed at a defined time over a defined period of time under a defined pressure.

The magnitude of the pressure exerted on the propellant during the metered addition is determined in particular in dependence on the required quantity of propellant, the type of article to be produced as well as the selected process parameters.

The present invention is explained in more detail below with reference to the figures on the basis of a preferred

embodiment by the example of the addition of a compressible fluid. It goes without saying that the following explanation may also be applied in principle to non-compressible fluids such as water, for example.

Figures 1a-1d show the individual stages of the process according to the invention for the production of physically foamed injection moulded articles;

Figure 2 schematically shows a device for executing the process according to the invention;

Figure 3 is a graph showing the pressure curve during execution of the process;

Figure 4 shows a variant of Figure 1 with direct introduction of the propellant into the cavity.

As Figure 1a shows, the cavity 1 of any injection moulding plant is partially initially filled in a first stage firstly with compact propellant-free melt 6. In this case, the feed pipe 3 for a compressed propellant is closed, for example, by a valve 4 such as a pressure relief valve (overflow valve).

After the cavity 1 has been filled with a desired quantity of propellant-free melt 6, the feed pipe 3 for the propellant is opened and the propellant is injected in compressed, preferably liquid, state via the injection point 5. Through contact with the hot melt, the liquid propellant turns to gas and expands under the lower pressure in the cavity.

As a general rule the propellant is still liquid and not gaseous at the injection point 5 itself, and therefore one cannot talk of a "gasification point" in a narrower sense.

The mixture 7 of gaseous propellant and melt flows into the cavity 1 and causes the cavity 1 to fill completely, in which

case the propellant-free melt portion 6 which was used for the initial filling comes to rest in the region of the cavity walls and forms the external skin or edge zone of the injection moulded article to be formed.

The cavity 1 can be ready filled as desired and required up to the maximum filling quantity with melt mixed with propellant or, as shown in Figure 1d, propellant-free melt can again be fed to the cavity in a third stage. In this case a foamed article is obtained which has a compact firm external skin right around which is formed by propellant-free melt.

After foaming and hardening, the finished injection moulded article, e.g. made of integral foam, is removed from the cavity and the cavity is immediately available again for the next charge.

As shown in Figure 1d, injection moulded articles, which have a cellular foamed internal core and a compact firm closed external skin, are obtained with the process according to the invention.

Contrary to the known foaming processes, such as those described above, in which the cavity is filled completely with a melt/propellant mixture, according to the invention an initial filling with propellant-free melt occurs firstly, as a result of which the formation of a uniform closed compact external skin is effected and articles with excellent surface characteristics can be obtained.

It is essential for execution of the process to prevent premature expansion of the propellant held under pressure. This can be achieved by appropriate insulation of the device and/or maintaining a suitable pressure level.

The metered addition of the propellant is conducted in a timeand pressure-controlled manner for the process according to the invention. Control can be carried out via a device which is also the subject of the invention.

As shown in Figure 2, the propellant stored under pressure in a storage means 11, e.g. a pressure cylinder etc., is fed to a pressure control valve 10, which can be a multi-way valve such as a 3/3- or 2/3-way proportional valve, and should advantageously have a very quick reaction time and precise regulation.

During the propellant injection phase, i.e. the phase in which the propellant is added to the melt, in the case of critical propellants, the compressed propellant passes via a pressure relief valve 4 to the injection point 5 and there is added to the melt.

In this case, the dimensions of the pipes, connection pieces and also the parts of the technical control system of the process are such that no premature expansion in volume of the propellant under pressure is possible.

In the case of a sudden increase in volume the aggregate state of the agent can change, i.e. the agent changes into a gas, in which case vaporisation cold is generated, which would in turn block the pipes as a result of "icing".

An increase in temperature on the way to the injection point 5 would also lead to a change in the aggregate state. For prevention, insulation of the heat-carrying elements is recommended.

In order to prevent premature expansion, all feed pipes should be as short as possible. Consequently, the pressure control valve 10 is preferably constructed to be as close as possible to the injection point 5. An improvement to the control characteristics of the control valve is also achieved as a result of the thus shortened feed pipe to the injection point 5. If critical propellants are used, a pressure relief valve or overflow valve 4 is provided before the injection point 5, this valve ensuring that the pressure in the device does not drop below a specific value, preferably p (crit) at the given temperature, at which the transformation of the propellant into gas would take place. If, for example, carbon dioxide is used as propellant, a pressure of at least 60 bar should be maintained at room temperature in order to keep the carbon dioxide in the device upstream in liquid state.

The pressure relief valve 4 ensures that the propellant remains in compressed state even during outage times of the machine, e.g. in the intermediate cycle times before and after or between the propellant injection phases. A full release of pressure only occurs when the machine or control system is switched off. Several pressure relief valves with "falling" pressure values may also be provided so that a pressure gradient is formed in front of the injection point 5 in the feed pipe section between the pressure control valve 10 and the pressure relief valve 4.

The graph in Figure 3 schematically shows the pressure curve for executing the process according to the invention using the example of compressible propellants.

Outside of the propellant injection phase, as in the intermediate cycle times, it is sufficient to keep the device at a selected pressure, at which the propellant respectively used remains in compressed, preferably liquid, state (section 20).

During the propellant injection phase (section 22), an elevated pressure is introduced in the feed pipes through the pressure control valve 10 so that the opening pressure (holding pressure) of the relief valve 4 is exceeded and the feed pipe section 3 up to the injection point 5 is quickly filled with liquid medium.

In this case, the pressure increase is proportional to the desired quantity of propellant to be fed to the melt. After time t expires, as soon as the desired quantity of propellant has been added to the melt, the pressure is reduced again to the starting pressure (section 24).

In Figure 3, sections 21 and 23 show the pressure build up or reduction phase.

The injection point 5 is preferably configured as a throttle means, e.g. as a defined gap in an injector, a sintered metal injector or a needle valve. A controlled closure mechanism is also particularly recommended for this. The quick pressure increase and the resistance through the injector prevent the propellant from transforming into gas, while the agent flows on from the pressure control valve 10.

The above measures ensure that the transformation of the agent into gas only occurs upon exit from the injector and when in contact with the hot melt, and that the inflowing melt is foamed.

After the propellant injection phase has ended, i.e. after the desired quantity of propellant has been added to the melt, the pressure in the feed pipe to the injection point 5 is reduced so that no propellant can flow on. However, in the pipe up to the pressure relief valve 4 the starting pressure remains in order to keep the agent in compressed or liquid state for the next cycle. A virtually pressure-free and thus gaseous state prevails only in the short feed pipe section from the pressure relief valve 4 to the injection point 5 until the next cycle. It goes without saying that this part of the plant may also be kept under pressure if required by the provision of a suitable closure mechanism which opens again at the beginning of the propellant injection phase as a result of the increasing pressure level.

The pressure control via the pressure control valve can occur automatically by providing pressure measurement points 12, 13, for example, in front of and behind the pressure control valve.

If carbon dioxide is used as propellant, for example, the plant is preferably held at an operating pressure of at least 60 bar at room temperature, so that the CO<sub>2</sub> also remains in compressed state during the periods between the propellant injection phases. At the beginning of the propellant injection phase, a desired working pressure of about 200 bar, for example, is built up (section 21) in order to assure an adequate flow of propellant to the melt. After the propellant injection phase 22 has ended, the pressure is reduced again to the desired operating pressure.

The injection point 5 is preferably located in the feeder pipe 3 close to the spray point x. According to a further embodiment, as is shown in Figure 4, the propellant can be added directly to the melt in the cavity. In this case, the injection point 5 is located directly at the cavity.

In addition, the build up of a counterpressure can be provided in the cavity 1, such as is also used in conventional injection moulding processes in the so-called gas counterpressure process.

Very short cycle times can be obtained with the process according to the invention. Hence, the process according to the invention is also very well suited to the production of mass-produced articles. The short cycle times are supported by the vaporisation cold resulting upon the transformation of the propellant into gas, and this causes a reduction in the cooling time, and thus also the cycle time.

Should there still be propellant residues present in the pore structure in the core of the article after demoulding, these slowly diffuse out of the article without detriment to its usability or recyclability.

Excellent dimensional stability of the article is achieved as a result of its closed firm external skin. In addition, foamed injection moulded articles which have a homogeneous uniform external skin and excellent tactility can be obtained with the process according to the invention.

The foamed injection moulded articles obtained have an excellent surface quality and do not require any further finishing. It is also of advantage that the cavity does not need to be treated with a separating agent.

The process according to the invention for the pressure-controlled metered addition of physical propellants to an expandable melt can be conducted advantageously with a device comprising a storage means 11, in which the propellant is stored under pressure, a pressure control valve 10 for regulating the propellant pressure and an injection point 5, which is preferably configured as a throttle means, at which the propellant under pressure is added to the melt, wherein in the case of critical propellants at least one pressure relief valve 4 is provided which is positioned downstream of the pressure control valve 10.

Although the above-described process and the device for the pressure-controlled metered addition of propellants under high pressure can be advantageously used for the production of physically foamed injection moulded articles, they are, of course, also suitable for other processes in which propellants are added under high pressure to melts to be expanded.

## List of Reference Numbers

1	cavity
2	melt feed
3	propellant feed pipe
4	pressure relief valve
5	injection point
6	propellant-free melt
7	melt with added propellant
8	injection of plastic material
9	mould comprising two halves
10	pressure control valve
11	propellant storage means
x	spray point

Section	20	pressure	during	the	intermediate	cycle
		times				
Section	21	pressure build up phase				
Section	22	propellar	nt injed	ction	n phase	
Section	23	pressure	reduct	ion p	phase	

#### Claims:

- 1. Process for the production of physically foamed injection moulded articles, characterised in that in a first stage a propellant-free first melt portion (6) is fed into a cavity (1) (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), wherein the dosage of the physical propellant occurs at least in a pressure regulated manner, and possibly in a third stage a propellant-free further melt portion is charged into the cavity (1).
- 2. Process according to Claim 1, characterised in that the propellant is a compressible fluid.
- 3. Process according to Claim 1 or 2, characterised in that the propellant is kept under pressure in the intermediate cycle times before and after the propellant injection phase, or is present in a compressed state.
- 4. Process according to Claim 3, characterised in that in the intermediate cycle times the propellant is held a pressure of at least p (crit) of the propellant at the given temperature.
- 5. Process according to one of the preceding claims, characterised in that the pressure exerted on the propellant is controlled via a pressure control valve (10).
- 6. Process according to Claim 5, characterised in that the pressure control valve (10) is a multi-way valve.
- 7. Process according to Claim 6, characterised in that a 3/3-way proportional valve or a 2/3-way proportional valve is used as multi-way valve.

- Process according to one of the preceding claims, characterised in that the pressure control in the case of critical propellants additionally occurs via at least one pressure relief valve (4) which is connected downstream of the pressure control valve (10).
  - 9. Process according to Claim 8, characterised in that the holding pressure of at least one of the pressure relief valves (4) is equal to or higher than the pressure at which a critical propellant is held in the intermediate cycle times.
  - 10. Process according to one of the preceding claims, characterised in that the pressure preset by the pressure control valve (10) is regulated via one or more pressure relief valves (4) to the injection pressure at which the propellant is added to the melt via an injection point (5).
  - 11. Process according to one of the preceding claims, characterised in that the injection point (5) is configured as a throttle means.
  - 12. Process according to Claim 11, characterised in that the injection point (5) is in the form of a defined gap in an injector or of an injector with a sinter metal.
  - 13. Process according to one of Claims 11 or 12, characterised in that the injection point (5) is configured as a controlled closure mechanism.
  - 14. Process according to Claim 1 or one of the preceding Claims 3 to 13, characterised in that water is used as propellant.
  - 15. Process according to one of the preceding Claims 1 to 13, characterised in that a gas or gas mixture is used as propellant.

- 16. Process according to Claims 15, characterised in that carbon dioxide is used as propellant.
- 17. Process according to Claims 16, characterised in that the carbon dioxide is held in the intermediate cycle times at a pressure of at least 60 bar (= p (crit)  $CO_2$  at room temperature).
- 18. Process according to one of the preceding claims, characterised in that for the propellant injection phase the propellant is brought to a pressure of over 60 bar via the pressure control valve (10).
- 19. Process according to one of the preceding claims, characterised in that a counterpressure is generated in the cavity (1).
- 20. Physically foamed injection moulded article with an external skin and a foamed core of the same material, characterised in that the foamed core is enclosed completely or partially by the external skin and the external skin is configured as a compact closed envelope without the addition of propellant.
- 21. Physically foamed injection moulded article according to Claim 20, characterised in that the injection moulded article is a handle, a knob, a gearshift knob, a steering wheel casing, a ball, a sphere, a fender or a float.
- 22. Physically foamed injection moulded article according to Claim 20, characterised in that the injection moulded article is a closing means for bottle-like containers such as a stopper or a cork.
- 23. Use of an injection moulded article according to one of Claims 20 or 21 in the automobile industry.

24. Device for the metered addition of physical propellants to a foamable melt, characterised in that the device comprises a storage means (11), in which the propellant is stored under pressure, a pressure control valve (10) for regulating the propellant pressure, and an injection point (5), which is configured as a throttle means, at which the propellant under pressure is fed to the melt, wherein in the case of critical propellants at least one pressure relief valve (4) is additionally provided downstream of the pressure control valve (10).

ENGLISH TRANSLATION OF APPLICIPATION SEP 172001

1 AS AMENDED

## Process for the Production of Physically Foamed Injection Moulded Articles

The present invention relates to a process for the production of physically foamed injection moulded articles, in particular injection moulded articles with an internal foam structure and a compact closed-pore external skin of the same material as the base body.

The production of foamed plastics, for example, is achieved with the aid of so-called propellants, which expand a plastic, generally thermally softened plastic mass in the desired manner. In this case, the propellants are either generated in situ via chemical reaction of the components (chemical propellants), or compressed fluids, e.g.  $N_2$ ,  $CO_2$ , are added under pressure to the starting material, in which case a foaming process of the plastic mass caused by the propellant is initiated upon the subsequent drop in pressure of the component mixture to normal pressure.

However, chemical propellants have a series of disadvantages. For instance, for use in foam injection moulding higher temperatures than are actually necessary for softening starting materials may have to be selected in order to reach the ignition point of the propellants, since the temperature at which the reaction of the components generating propellant starts is generally very high. Because of the high temperatures a higher expenditure of energy is necessary during melting of the raw materials. In addition, the cycle or cooling times are increased and a higher cooling power of the cooling plants is necessary. In some circumstances, damage to the raw materials may also occur as a result of the comparatively high temperatures.

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Chemical propellants which have not been converted can locate on the surface of the articles obtained and cause yellowing of the articles. Allergic skin reactions may also result upon contact with these articles.

Foam articles which have been obtained by means of chemical propellants are not recyclable, or if so only conditionally, since there is the risk that non-ignited propellants can lead to uncontrolled reactions during reuse.

Therefore, physical propellants are preferably used to foam plastics. Physical propellants allow optimum adaptation of the melting temperature to the respectively selected raw material, as a result of which the energy expenditure is reduced, optimum cycle and cooling times are made possible and in addition there is no risk that the raw materials could be detrimentally affected as a result of temperatures which are too high. Moreover, inexpensive gases such as  $CO_2$ , for example, can be used as physical propellants.

Physical propellants do not remain in the finished foam articles, but diffuse out within a comparatively short time. Therefore, these articles are fully recyclable, since there is no need to fear that propellant residues could lead to uncontrolled reactions.

Various processes are known for the production of articles from foamed plastic with a compact closed external skin and a cellular core cohering with the external skin or edge zone, also referred to as integral foam or structural foam.

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For example, in the reaction injection moulding process (RIM), two reactive components are mixed together which harden and foam in the cavity of a mould under reaction. Because of the quicker cooling at the wall of the mould, the reaction mass solidifies more quickly there than in the interior of the mould, and as a result the foaming process ceases earlier there than in the mould interior, and a compact sealed external layer is formed.

As determined by the process, the reactive component mixture must be comparatively liquid in order to guarantee complete filling of the mould before the reaction starts. However, this leads to irregularities on the surface of the formed article as a result of spray over and skin formation, which necessitates expensive finishing for high-grade articles, for which a perfect surface is required.

Moreover, for the RIM process the mould must be treated with a separating agent prior to injection, which on the one hand requires more expenditure in processing and can additionally lead to residues on the finished article which must be removed. The relatively long cycle times are also disadvantageous.

Since foaming in the RIM process is generally conducted chemically, the articles to be obtained are only conditionally recyclable.

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Integral foams made of polyurethane to be used as working material primarily in the automobile industry, e.g. for steering wheel casings or gearshift knobs etc., are preferably produced using the RIM process. However, for this field of application the articles must not only have as perfect a surface as possible, but also have pleasant skin feel (tactility).

It has been shown that articles of polyurethane integral foam have only a conditionally acceptable tactility.

5 It is also known to produce integral foams from thermoplastic urethane or thermoplastic elastomer by means of conventional moulding processes. Both chemical and propellants can be used in this case. Contrary to the RIM process, which requires special plants, already existing injection moulding 10 plants without expensive refitting can be used for this. The state of the s

The necessary finishing of the articles obtained is only slight.

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DE 196 46 665 Al describes a process for metering physical propellants, wherein a propellant is added at high pressure to the softened plastic material transported in the consumer, e.g. an extruder or an RIM machine, and the amount of propellant is regulated with a pressure control valve, which keeps the pressure difference constant via a rigid throttle means by regulating the pressure difference in dependence on the flow of propellant. The extrusion processes described here are continuous processes in which the propellant is permanently added.

A process for the production of multilayered articles with a foamed 25 core and a non-expanded thermoplastic external skin is known from DE 1 778 457, wherein a first propellant-free melt and a second melt containing propellant as well as possibly a third propellantfree melt are firstly prepared and injected one after the other into an appropriate mould, in which case the mould must be 30 maintained at a temperature equal to or higher than the activation temperature of the propellant.

Where physical propellants are used, it is suggested that either the selected temperature of the melt upon leaving the nozzle is so high that, when a mould with constant internal volume is used, the gas formation, and thus the expansion, still occurs below the pressure exerted on the substance in the mould, and when a mould with extendable interior is used, the gas formation, and thus the expansion, occurs by relieving the pressure exerted on the mould interior to expand the mould. There is no mention of the propellant being added directly to the melt flow which flows into the mould, nor of the quantity of propellant apportioned to the melt flow being regulated via the pressure.

An improved process of the aforementioned type is specified in DE 1 948 454, wherein the propellant is injected into the melt flow shortly before entry into the mould and the injection period is continued until the mixture quantity required to form the core has been inserted into the mould. Solvents with a boiling point preferably between 20 and 150°C are specified as propellants, which are to prevent premature expansion under a corresponding pressure. There is likewise no mention here of a pressure regulation of the added quantity of propellant to the melt.

A process for the production of injection moulded articles with foamed core is described in the US patent 4,548,776, according to which gaseous of gas-generated chemical propellant is already added to the melt in the extruder, is thoroughly mixed with this and the already foamed melt is then injected into the mould.

In this case, the addition of propellant occurs via a porous insert at the injection point, a supply valve being provided in the feed

The object of the present invention is to provide a process for the production of physically foamed injection moulded articles, with which injection moulded articles with an integral structure, excellent surface characteristics, thus rendering expensive finishing unnecessary, and additionally excellent tactility, can be obtained in a simple manner using conventional injection moulding plants.

The articles produced according to the invention are suitable in particular for fields of application which set high quality requirements for surface structure and for which a pleasant sensory feel is of advantage on skin contact. The automobile industry is given as an example, for which handles, knobs such as gearshift knobs, steering wheel casings etc. of the foamed plastics obtained according to the invention can be used. However, the process according to the invention is in no way restricted to the production of articles for the automobile industry, but is quite generally suitable for the production of any desired foamed injection moulded articles.

For example, mass-produced articles such as closing means for bottle-like containers, e.g. stoppers or corks, may also be advantageously obtained according to this process. Further examples are balls, spheres, fenders, floats etc.

A further field of use is the production of supporting parts, for example, for the aviation or automobile industry, in particular for parts where strength is relevant.

5 This object is achieved according to the invention by a process for the production of physically foamed injection moulded articles, wherein firstly in a first stage a propellant-free first melt portion is fed into a cavity (initial filling), in a second stage a physical propellant is added at elevated pressure to the 10 following melt portion (propellant injection phase), wherein there is the first first first first metering of the physical propellant occurs at least in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure which is exerted on the propellant in the phases between or before or after metered addition, and the expansion of the propellant occurs in the cavity, and possibly in a third stage a propellant-free further melt portion is charged into the cavity.

This process also permits the formation of physically foamed injection moulded articles, the foamed core of which is completely or partially enclosed by a compact closed external skin, which has been produced without the addition of propellants, the core and the external skin being made of the same material.

25 The present invention additionally relates to a device for the metered addition of propellants under elevated pressure to an expandable melt.

This device can also be advantageously used for the metered addition of compressible propellants. 30

The propellant-free melt portion firstly fed into the cavity in the first stage forms a compact closed external skin without pores in the finished foamed injection moulded articles.

Any desired fluid which expands upon corresponding pressure relief and foams the melt material in a suitable manner can be used as propellant. Hence, compressible fluids such as gases in liquid or supercritical phase, for example, may be used.

The use of carbon dioxide is recommended because of its ready availability.

A further preferred propellant is water.

The starting material for the melt is not subject to any special restrictions. Any desired thermoplastic melt material which is suitable for injection moulding and can be foamed may be used.

Examples are thermoplastic materials, but also further thermoplastic melts, such as metallic or ceramic melts, for example. Examples of metallic materials include aluminium, magnesium, zinc, tin or even precious metals.

The process according to the invention leads to weight reduction and strength increase in comparison to the corresponding compact articles.

"Pressure regulated" in the sense of the invention means that in the course of the process the pressure exerted on the propellant is varied for metered addition of the propellant.

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In this case the pressure exerted on the propellant during the propellant injection phase is greater than the pressure exerted on the propellant in the phases between or before or after metered addition. This means in the case of critical or compressible propellants, for example, that the pressure exerted in the intermediate cycle times is lower than the holding pressure of a pressure relief valve or overflow valve.

Therefore, according to the invention the required proportion of propellant is added to a melt to be foamed at a defined time over a defined period of time under a defined pressure.

The magnitude of the pressure exerted on the propellant during the metered addition is determined in particular in dependence on the required quantity of propellant, the type of article to be produced as well as the selected process parameters.

The present invention is explained in more detail below with reference to the figures on the basis of a preferred embodiment by the example of the addition of a compressible fluid. It goes without saying that the following explanation may also be applied in principle to non-compressible fluids such as water, for example.

Figures 1a-1d show the individual stages of the process according to the invention for the production of physically foamed injection moulded articles;

Figure 2 schematically shows a device for executing the process according to the invention;

Figure 3 is a graph showing the pressure curve during execution of the process;

Figure 4 shows a variant of Figure 1 with direct introduction of the propellant into the cavity.

As Figure 1a shows, the cavity 1 of any injection moulding plant is partially initially filled in a first stage firstly with compact propellant-free melt 6. In this case, the feed pipe 3 for a compressed propellant is closed, for example, by a valve 4 such as a pressure relief valve (overflow valve).

After the cavity 1 has been filled with a desired quantity of propellant-free melt 6, the feed pipe 3 for the propellant is opened and the propellant is injected in compressed, preferably liquid, state via the injection point 5. Through contact with the hot melt, the liquid propellant turns to gas and expands under the lower pressure in the cavity.

As a general rule the propellant is still liquid and not gaseous at the injection point 5 itself, and therefore one cannot talk of a "gasification point" in a narrower sense.

The mixture 7 of gaseous propellant and melt flows into the cavity 1 and causes the cavity 1 to fill completely, in which case the propellant-free melt portion 6 which was used for the initial filling comes to rest in the region of the cavity walls and forms the external skin or edge zone of the injection moulded article to be formed.

The cavity 1 can be ready filled as desired and required up to the maximum filling quantity with melt mixed with propellant or, as shown in Figure 1d, propellant-free melt can again be fed to the cavity in a third stage. In this case a foamed article is obtained which has a compact firm external skin right around which is formed by propellant-free melt.

After foaming and hardening, the finished injection moulded article, e.g. made of integral foam, is removed from the cavity and the cavity is immediately available again for the next charge.

As shown in Figure 1d, injection moulded articles, which have a cellular foamed internal core and a compact firm closed external skin, are obtained with the process according to the invention. Contrary to the known foaming processes, such as those described above, in which the cavity is filled completely with a melt/propellant mixture, according to the invention an initial filling with propellant-free melt occurs firstly, as a result of which the formation of a uniform closed compact external skin is effected and articles with excellent surface characteristics can be obtained.

It is essential for execution of the process to prevent premature expansion of the propellant held under pressure. This can be achieved by appropriate insulation of the device and/or maintaining a suitable pressure level.

The metered addition of the propellant is conducted in a time- and pressure-controlled manner for the process according to the invention. Control can be carried out via a device which is also the subject of the invention.

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As shown in Figure 2, the propellant stored under pressure in a storage means 11, e.g. a pressure cylinder etc., is fed to a pressure control valve 10, which can be a multi-way valve such as a 3/3- or 2/3-way proportional valve, and should advantageously have a very quick reaction time and precise regulation.

During the propellant injection phase, i.e. the phase in which the propellant is added to the melt, in the case of critical propellants, the compressed propellant passes via a pressure relief valve 4 to the injection point 5 and there is added to the melt.

In this case, the dimensions of the pipes, connection pieces and also the parts of the technical control system of the process are such that no premature expansion in volume of the propellant under pressure is possible.

In the case of a sudden increase in volume the aggregate state of the agent can change, i.e. the agent changes into a gas, in which case vaporisation cold is generated, which would in turn block the pipes as a result of "icing".

An increase in temperature on the way to the injection point 5 would also lead to a change in the aggregate state. For prevention, insulation of the heat-carrying elements is recommended.

In order to prevent premature expansion, all feed pipes should be as short as possible. Consequently, the pressure control valve 10 is preferably constructed to be as close as possible to the injection point 5. An improvement to the control characteristics of

the control valve is also achieved as a result of the thus shortened feed pipe to the injection point 5.

If critical propellants are used, a pressure relief valve or overflow valve 4 is provided before the injection point 5, this valve ensuring that the pressure in the device does not drop below a specific value, preferably p (crit) at the given temperature, at which the transformation of the propellant into gas would take place. If, for example, carbon dioxide is used as propellant, a pressure of at least 60 bar should be maintained at room temperature in order to keep the carbon dioxide in the device upstream in liquid state.

The pressure relief valve 4 ensures that the propellant remains in compressed state even during outage times of the machine, e.g. in the intermediate cycle times before and after or between the propellant injection phases. A full release of pressure only occurs when the machine or control system is switched off. Several pressure relief valves with "falling" pressure values may also be provided so that a pressure gradient is formed in front of the injection point 5 in the feed pipe section between the pressure control valve 10 and the pressure relief valve 4.

The graph in Figure 3 schematically shows the pressure curve for executing the process according to the invention using the example of compressible propellants.

Outside of the propellant injection phase, as in the intermediate cycle times, it is sufficient to keep the device at a selected pressure, at which the propellant respectively used remains in compressed, preferably liquid, state (section 20).

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During the propellant injection phase (section 22), an elevated pressure is introduced in the feed pipes through the pressure control valve 10 so that the opening pressure (holding pressure) of the relief valve 4 is exceeded and the feed pipe section 3 up to the injection point 5 is quickly filled with liquid medium.

In this case, the pressure increase is proportional to the desired quantity of propellant to be fed to the melt. After time t expires, as soon as the desired quantity of propellant has been added to the melt, the pressure is reduced again to the starting pressure (section 24).

In Figure 3, sections 21 and 23 show the pressure build up or reduction phase.

The injection point 5 is preferably configured as a throttle means, e.g. as a defined gap in an injector, a sintered metal injector or a needle valve. According to the invention, a controlled closure mechanism is located at the injection point. The quick pressure increase and the resistance through the injector prevent the propellant from transforming into gas, while the agent flows on from the pressure control valve 10.

The above measures ensure that the transformation of the agent into gas only occurs upon exit from the injector and when in contact with the hot melt, and that the inflowing melt is foamed.

The controlled closure mechanism can be omitted if a pressure relief valve is provided.

After the propellant injection phase has ended, i.e. after the desired quantity of propellant has been added to the melt, the pressure in the feed pipe to the injection point 5 is reduced so that no propellant can flow on. However, in the pipe up to the pressure relief valve 4 the starting pressure remains in order to keep the agent in compressed or liquid state for the next cycle. A virtually pressure-free and thus gaseous state prevails only in the short feed pipe section from the pressure relief valve 4 to the injection point 5 until the next cycle.

It goes without saying that this part of the plant may also be kept under pressure if required by the provision of a suitable closure mechanism which opens again at the beginning of the propellant injection phase as a result of the increasing pressure level.

The pressure control via the pressure control valve can occur automatically by providing pressure measurement points 12, 13, for example, in front of and behind the pressure control valve.

If carbon dioxide is used as propellant, for example, the plant is preferably held at an operating pressure of at least 60 bar at room temperature, so that the  ${\rm CO_2}$  also remains in compressed state during the periods between the propellant injection phases. At the beginning of the propellant injection phase, a desired working pressure of about 200 bar, for example, is built up (section 21) in order to assure an adequate flow of propellant to the melt. After the propellant injection phase 22 has ended, the pressure is reduced again to the desired operating pressure.

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The injection point 5 is preferably located in the feeder pipe 3 close to the spray point x. According to a further embodiment, as is shown in Figure 4, the propellant can be added directly to the melt in the cavity. In this case, the injection point 5 is located directly at the cavity.

In addition, the build up of a counterpressure can be provided in the cavity 1, such as is also used in conventional injection moulding processes in the so-called gas counterpressure process.

Very short cycle times can be obtained with the process according to the invention. Hence, the process according to the invention is also very well suited to the production of mass-produced articles. The short cycle times are supported by the vaporisation cold resulting upon the transformation of the propellant into gas, and this causes a reduction in the cooling time, and thus also the cycle time.

Should there still be propellant residues present in the pore structure in the core of the article after demoulding, these slowly diffuse out of the article without detriment to its usability or recyclability.

Excellent dimensional stability of the article is achieved as a result of its closed firm external skin. In addition, foamed injection moulded articles which have a homogeneous uniform external skin and excellent tactility can be obtained with the process according to the invention.

The foamed injection moulded articles obtained have an excellent surface quality and do not require any further finishing. It is

also of advantage that the cavity does not need to be treated with a separating agent.

The process according to the invention for the pressure-controlled metered addition of physical propellants to an expandable melt can be conducted advantageously with a device comprising a storage means 11, in which the propellant is stored under pressure, a pressure control valve 10 for regulating the propellant pressure and an injection point 5, which is preferably configured as a throttle means, at which the propellant under pressure is added to the melt, wherein the injection point 5 includes a controlled closure mechanism, and in the case of critical propellants at least one pressure relief valve 4 is provided which is positioned downstream of the pressure control valve 10.

Although the above-described process and the device for the pressure-controlled metered addition of propellants under high pressure can be advantageously used for the production of physically foamed injection moulded articles, they are, of course, also suitable for other processes in which propellants are added under high pressure to melts to be expanded.

# List of Reference Numbers

	1	cavity				
5	2	melt feed				
	3	propellant feed pipe				
	4	pressure relief valve				
	5	injection	injection point			
10 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	6	propellan	propellant-free melt			
	7	melt with added propellant				
	8	injection of plastic material				
	9	mould comp	prising two halves			
**!	10	pressure	control valve			
	11	propellant storage means				
15=						
<b>T</b>	x	spray poi	nt			
12.j						
	Section	20	pressure during the intermediate cycle			
20			times			
	Section	21	pressure build up phase			
	Section	22	propellant injection phase			
	Section	23	pressure reduction phase			
	Section	23	pressure reduction phase			

#### Claims:

1. Process for the production of physically foamed injection moulded articles, wherein in a first stage a propellant-free first melt portion (6) is fed into a cavity (1) (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), and possibly in a third stage a propellant-free further melt portion is charged into the cavity (1), the production of the injection moulded articles occurring in the cavity,

characterised in that metering of the physical propellant in the second stage occurs in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure which is exerted on the propellant in the phases between or before or after metered addition, and the expansion of the propellant occurs in the cavity (1).

- 2. Process according to Claim 1, characterised in that the propellant is a compressible fluid.
- 3. Process according to Claim 1 or 2, characterised in that the propellant is kept under pressure in the intermediate cycle times before and after the propellant injection phase, or is present in a compressed state.
- 4. Process according to Claim 3, characterised in that in the intermediate cycle times the propellant is held a pressure of at least p (crit) of the propellant at the given temperature.

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- 5. Process according to one of the preceding claims, characterised in that the pressure exerted on the propellant is controlled via a pressure control valve (10).
- 5 6. Process according to Claim 5, characterised in that the pressure control valve (10) is a multi-way valve.
  - 7. Process according to Claim 6, characterised in that a 3/3-way proportional valve or a 2/3-way proportional valve is used as multi-way valve.
  - 8. Process according to one of the preceding claims, characterised in that the pressure control in the case of critical propellants additionally occurs via at least one pressure relief valve (4) which is connected downstream of the pressure control valve (10).
  - 9. Process according to Claim 8, characterised in that the holding pressure of at least one of the pressure relief valves (4) is equal to or higher than the pressure at which a critical propellant is held in the intermediate cycle times.
- 10. Process according to one of the preceding claims, characterised in that the pressure preset by the pressure control valve (10) is regulated via one or more pressure relief valves (4) to the injection pressure at which the propellant is added to the melt via an injection point (5).
- 11. Process according to one of the preceding claims, characterised in that the injection point (5) is configured as a throttle means.

- 12. Process according to Claim 11, characterised in that the injection point (5) is in the form of a defined gap in an injector or of an injector with a sinter metal.
- 5 13. Process according to one of Claims 11 or 12, characterised in that the injection point (5) is configured as a controlled closure mechanism.
- 14. Process according to Claim 1 or one of the preceding Claims 3 to 13, characterised in that water is used as propellant.
  - 15. Process according to one of the preceding Claims 1 to 13, characterised in that a gas or gas mixture is used as propellant.
  - 16. Process according to Claims 15, characterised in that carbon dioxide is used as propellant.

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- 17. Process according to Claims 16, characterised in that the carbon dioxide is held in the intermediate cycle times at a pressure of at least 60 bar (= p (crit)  $CO_2$  at room temperature).
- 18. Process according to one of the preceding claims, 25 characterised in that for the propellant injection phase the propellant is brought to a pressure of over 60 bar via the pressure control valve (10).
- 19. Process according to one of the preceding claims, characterised in that a counterpressure is generated in the cavity (1).
  - 20. Process according to one of the preceding claims,

characterised in that the physically foamed injection moulded article is selected from a handle, a knob, a gearshift knob, a steering wheel casing, a ball, a sphere, a fender, a float and a closing means for bottle-like containers.

21. Device for the metered addition of physical propellants to a foamable melt, wherein the device comprises a storage means (11), in which the propellant is stored under pressure, a pressure control valve (10) for regulating the propellant pressure, and an injection point (5), which is configured as a throttle means, at which the propellant under pressure is fed to the melt, characterised in that a controlled closure mechanism is

characterised in that a controlled closure mechanism is provided at the injection point (5).

22. Device for the metered addition of physical propellants according to Claim 21, characterised in that instead of the controlled closure mechanism or in addition to the controlled closure mechanism, at least one pressure relief valve (4) is provided.

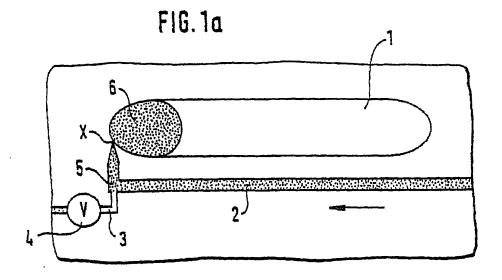


FIG.1a

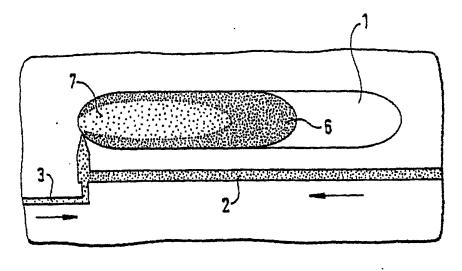


FIG.1b

FIG. 1b

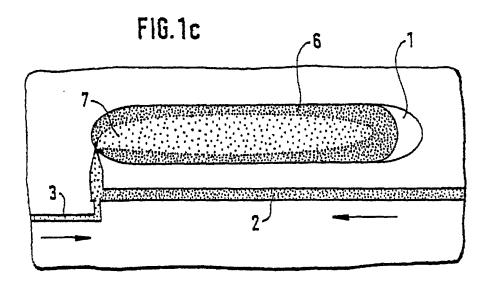


FIG. 1c

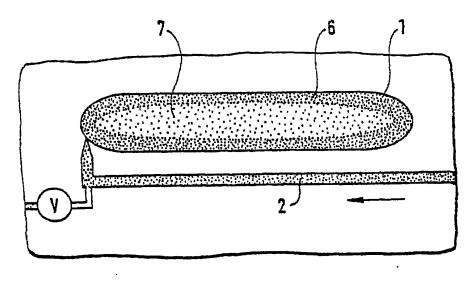
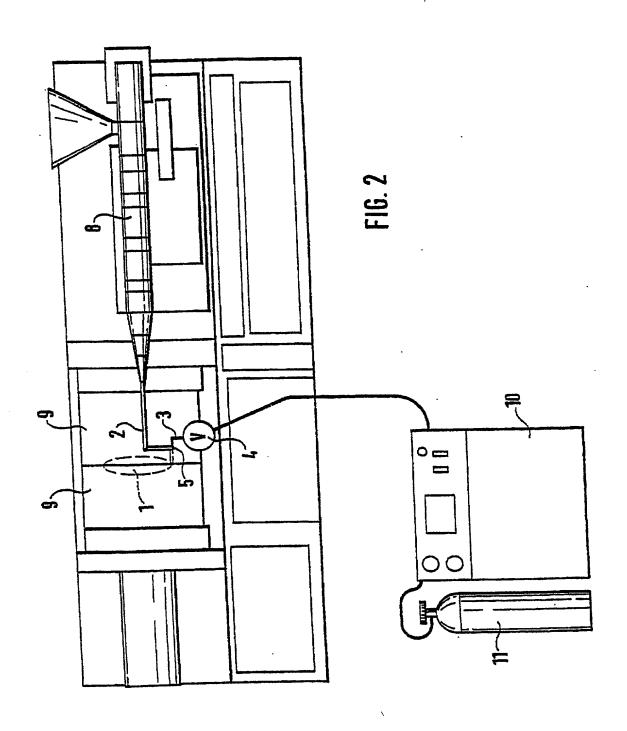


FIG.-1d

FIG.1d



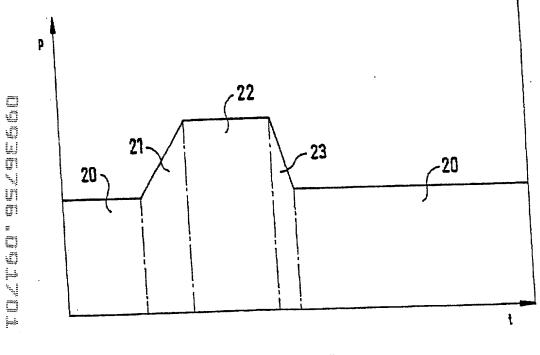
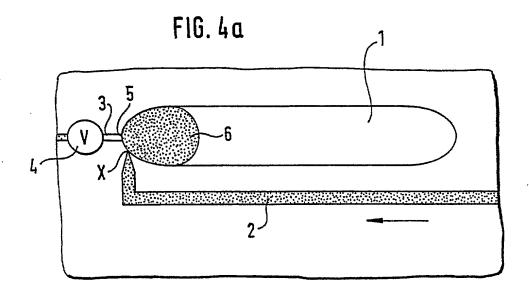


FIG. 3

F1G. 3



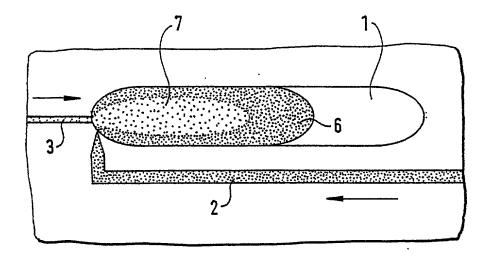


FIG. 4b

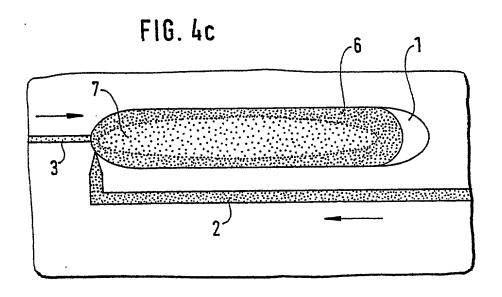


FIG. 4c

FIG. 4d

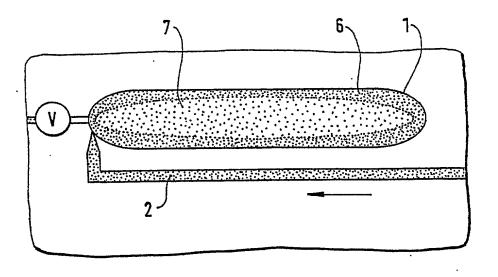


FIG. 4d

#### COMBINED DECLARATION AND POWER OF ATTORNEY

# (ORIGINAL, DESIGN, NATIONAL STAGE OF PCT, SUPPLEMENTAL, DIVISIONAL, CONTINUATION, OR C-I-P)

As a below named inventor, I hereby declare that:

#### TYPE OF DECLARATION

This declaration is for a national stage of PCT application.

#### INVENTORSHIP IDENTIFICATION

My residence, post office address and citizenship are as stated below, next to my name. I believe that I am the original, first and sole inventor of the subject matter that is claimed, and for which a patent is sought on the invention entitled:

#### TITLE OF INVENTION

# METHOD FOR PRODUCING PHYSICALLY FOAMED INJECTION MOULDED PARTS

#### SPECIFICATION IDENTIFICATION

The specification was described and claimed in PCT International Application No. PCT/EP00/02258 filed on March 15, 2000.

# ACKNOWLEDGMENT OF REVIEW OF PAPERS AND DUTY OF CANDOR

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information, which is material to patentability as defined in 37, Code of Federal Regulations, Section 1.56.

### PRIORITY CLAIM (35 U.S.C. Section 119(a)-(d))

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed.

Such applications have been filed as follows.

(Declaration and Power of Attorney-page 1 of 3)

# PRIOR PCT APPLICATION(S) FILED WITHIN 12 MONTHS (6 MONTHS FOR DESIGN) PRIOR TO THIS APPLICATION AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. SECTION 119(a)-(d)

INDICATE IF PCT	APPLICATION NUMBER	DATE OF FILING DAY, <b>MONTH</b> , YEAR	PRIORITY CLAIMED UNDER 35 U.S.C. SECTION 119
PCT	PCT/EP00/02258	15 March 2000	yes

## POWER OF ATTORNEY

I hereby appoint the following practitioner(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

APPOINTED PRACTITIONER(S)

REGISTRATION NUMBER(S)

Jodi-Ann McLane

Michele J. Young

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I hereby appoint the practitioner(s) associated with the Customer Number provided below to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith.

SEND CORRESPONDENCE TO

**DIRECT TELEPHONE CALLS TO:** 

Jodi-Ann McLane 401-421-3141

Jodi-Ann McLane
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US

Customer Number 000987

DECLARATION

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

SIGNATURE(S)

1-00	Ulrich STIELER Inventor's signature		Ulrich St	heles	
	Date	2001	Country of Citi	zenship Germany	-
	Residence Goslar	Germany			
	Post Office Address	Fontanewe	1. Goslar D-38642 German	ıv	

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